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Reference Method for Source Testing: Measurement of Releases of Fine Particulate Matter from Stationary Sources

Stationary Sources
Emissions Research & Measurement Section
Science & Technology Branch
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Gatineau QC K1A 0H3
Telephone: 819-997-2800
Toll Free: 1-800-668-6767 (in Canada only)
Fax: 819-994-1412
TTY: 819-994-0736
Email: enviroinfo@ec.gc.ca

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Foreword

The test methods presented in this report (G for $PM_{2.5}$ and H for Condensable Particulate Matter) may be used separately or jointly to determine fine filterable and condensable particulate matter. These methods are used with Method E of Reference Method EPS 1/RM/8 for the measurement of releases of particulate from stationary sources. The filterable particulate matter (+$PM_{2.5}$ and $PM_{2.5}$) and condensable particulate matter measured in this method are considered primary or direct particulate matter.

"Must" and "Shall" are used to indicate that the instructions in this method are mandatory. "Should" is used to indicate that a provision in this method is not an absolute requirement but is highly recommended as good practice. "May" is used to mean an allowance or option or to indicate a certain measure of likelihood or possibility.

The complexity of these methods warrants that personnel performing them be trained and experienced. The application of this method will entail health and safety hazards. Sampling organizations and their staff are responsible for obtaining the required training to meet standards for occupational health and safety, complying with the transportation of dangerous goods and developing site-specific health and safety plans.

Note: Mention of trade names or commercial products does not constitute endorsement for use by Environment Canada.

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Method G: Determination of Filterable $PM_{2.5}$ and Filterable Particulate Matter

1. Applicability

This method is used to measure the mass concentration and mass emission or release of filterable aerodynamic $PM_{2.5}$ and filterable particulate matter (PM) from enclosed gas streams of stationary sources. The filterable PM is considered equivalent to the particulate matter from Method E, Report EPS 1/RM/8.

Direct application of the procedures specified in this method may be limited by one or more of the following conditions:

- sample locations less than two stack diameters downstream or less than 0.5 stack diameter upstream of a flow disturbance;
- duct cross-sectional areas less than 0.071 m^2 (113 in^2) or duct diameters less than 0.3 m (12 in);
- supersaturated gas streams with entrained water droplets;
- gas stream velocities less than 3 m/s (10 ft/s) or greater than 30 m/s (100 ft/s);
- stack gas temperatures higher than 260°C (500°F) as they may cause damage to the $PM_{2.5}$ cyclone;
- flammable or explosive gas streams, or those containing corrosive or unstable components;
- sources with such high particulate levels that would affect the $PM_{2.5}$ separation;
- cyclonic flow patterns within the gas stream; or
- rapid fluctuations in velocity, moisture, particulate loading, and/or temperature of the gas stream due to uncontrollable process variations.

For compliance testing, possible modifications to allow sampling of sources exhibiting any of these characteristics must be approved in writing by Environment Canada.

2. Principle

Particulate matter is withdrawn at near-constant rate, but isokinetically, from pre-determined traverse points in an enclosed gas stream. The filterable $PM_{2.5}$ is separated with an in-stack cyclone, and deposited in the probe and on an out-of stack heated filter maintained at a temperature of 120 ± 14°C (248 ± 25°F) or at such other temperature as is necessary to prevent blinding of the filter from condensation. The filterable $PM_{2.5}$ and filterable PM is determined gravimetrically after removal of uncombined water. Simultaneous determinations of the gas stream moisture content, velocity, temperature and molecular weight allow the calculation of $PM_{2.5}$ concentration and emission rate. This method is used in conjunction with the Methods of Report EPS 1/RM/8, December 1993, *Standard Reference Method for Source Testing: Measurement of Releases of Particulate from Stationary Sources.*

Sampling isokinetically means that the velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sampling point.

Three valid tests are required for the determination of filterable $PM_{2.5}$ and particulate matter. Each test must be a minimum of two hours and collect at least 1.5 m^3 (53 ft^3) of stack gas on a dry basis at reference conditions.

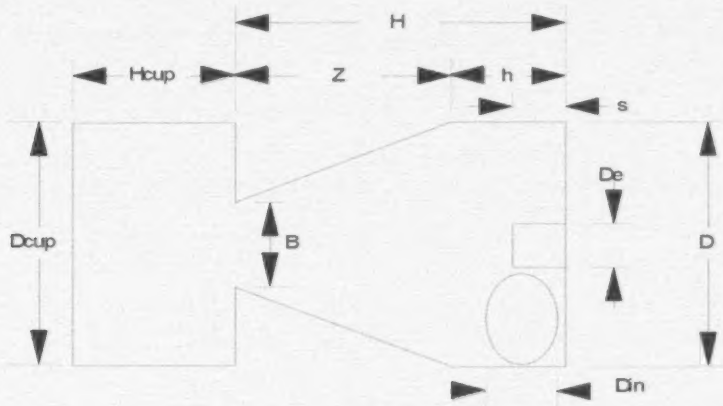
3. Apparatus

3.1 Sample Collection

The following items are required for sample collection:

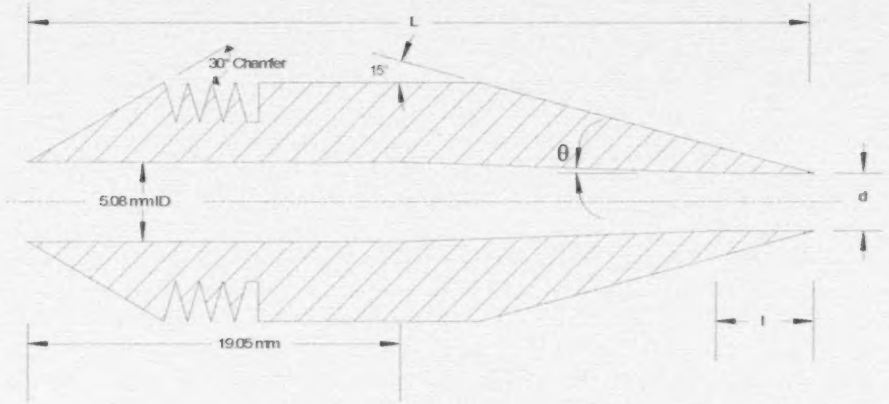
$PM_{2.5}$ Cyclone. A stainless steel (316 or equivalent) or Teflon-coated $PM_{2.5}$ cyclone that meets the dimensional specifications of Figure G-1 to within ± 0.02 cm (± 0.01 in). The fluoropolymer O-rings used in the cyclone have a temperature limit of approximately 205°C (400°F). Use stainless steel sealing rings for temperatures between 205°C and 260°C (400°F to 500°F). Contact Environment Canada for temperatures above 260°C (500°F).

Nozzles. A set of straight stainless steel nozzles (316 or equivalent) with sharp, tapered leading edges, as shown in Figures G-2 and G-3. The nozzles may be coated with Teflon.



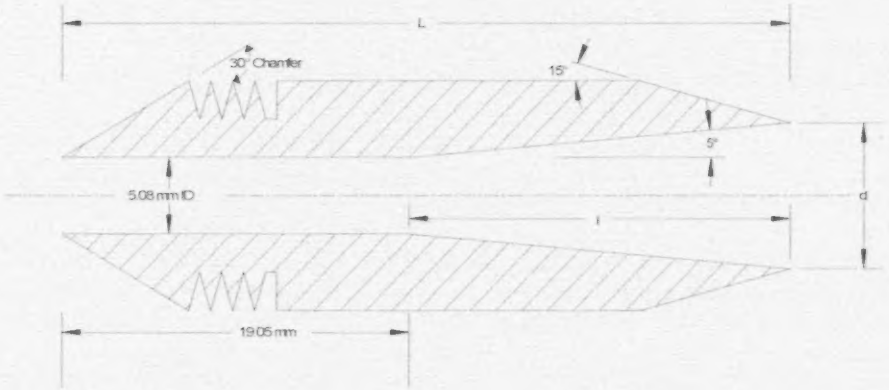
Cyclone IV (2.5 micrometer)	Cyclone Interior Dimensions (cm ± 0.02 cm)									
	D_{in}	D	D_e	B	H	h	Z	S	H_{cup}	D_{cup}
	0.51	2.54	0.59	1.09	2.68	1.03	1.65	0.58	2.22	2.62

Figure G-1 Cyclone Interior Dimensions



Nozzle Diameter, d (mm)	Internal Cone Angle, θ (degrees)	Straight Inlet Length, l (mm)	Total Length, L (mm)
3.175	3	≤ 1.27	36.830 ± 1.27
3.505	2	≤ 1.27	36.830 ± 1.27
3.962	1	≤ 1.27	36.830 ± 1.27
4.369	1	≤ 1.27	36.830 ± 1.27
4.775	1	≤ 1.27	36.830 ± 1.27
5.080	0	≤ 1.27	36.830 ± 1.27

Figure G-2 Nozzle Design for $PM_{2.5}$ Cyclone (High Stack Gas Velocity)



Nozzle Diameter, d (mm)	Straight Inlet Length, l (mm)	Total Length, L (mm)
5.486	2.362	21.412
5.944	4.928	23.978
6.426	7.722	26.772
6.960	10.719	29.769
7.518	13.945	32.995
8.128	17.475	36.525

Figure G-3 Nozzle Design for $PM_{2.5}$ Cyclone (Low Stack Gas Velocity)

An example of an assembled $PM_{2.5}$ cyclone and nozzle assembly is illustrated in Figure G-4. This assembly will pass through a standard 102 mm (4 in) port.

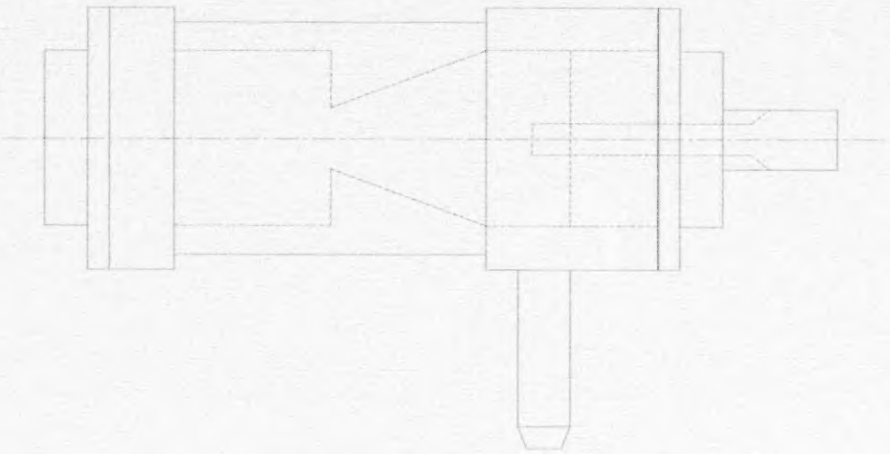


Figure G-4 Assembled $PM_{2.5}$ Cyclone

Probe. A borosilicate or quartz glass liner. Where length or strength limitations preclude the use of a glass liner, a seamless tubing made from an inert and corrosion-resistant material such as 316 stainless steel, Incoloy 825 or Teflon may be used. The liner is encased in a stainless-steel tube with an outer diameter equal to 2.5 cm (1.0 in) and a heating and temperature indicating system capable of maintaining the exit gas temperature at 120 ± 14°C (248 ± 25°F), or at such a temperature necessary to prevent condensation.

Pitot Tube. An S-type (Stausscheibe) pitot tube attached to the probe. The face openings of the pitot tube and the cyclone nozzle shall be parallel to each other. The pitot tube must be separated from the $PM_{2.5}$ cyclone and the temperature sensor by at least 2.5 cm (1.0 in). The probe assembly shall be calibrated according to the procedures specified in Reference Method EPS 1/RM/8, Method F. The calibration must be performed with the entire sampling assembly (cyclone, pitot tube and thermocouple).

Stack Temperature Sensor. A calibrated thermocouple or other suitable temperature sensor capable of measuring the stack temperature within 1.5% of the minimum absolute stack temperature. The sensor must be separated from the $PM_{2.5}$ cyclone and the pitot tube by at least 2.5 cm (1.0 in).

Primary Filter Holder. A borosilicate filter holder with an inert filter support (glass frit, Teflon plate, perforated stainless steel mesh or perforated plate) fitted with soft sealing surfaces to prevent gas leakage into or around the filter. A Teflon filter support with fluoropolymer O-rings is recommended. The filter must not be pinched between the O-ring and the filter support.

The primary filter holder is located inside the filter compartment.

Filter Compartment Heating System. A heating system capable of maintaining the temperature in the filter compartment at 120 ± 14°C (248 ± 25°F) or a higher temperature as is necessary to prevent blinding of the filter from condensation. A thermocouple or other temperature sensor is also required to measure the compartment temperature to within 3°C (5°F).

Impingers. Four Greenburg-Smith impingers are connected in series. The first, third and fourth impingers are modified by replacing the tips and impaction plates of the standard design with a 13 mm (0.5 in) ID glass tube extending to within 13 mm (0.5 in) of the bottom of the impinger. The second impinger has the standard tip and impaction plate. The impingers are contained in an ice bath during sampling. The impingers may be replaced by any other suitable condenser providing that the condensed liquid is to be used for moisture determination only. A temperature sensor capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger.

Vacuum Pump. A leakless vacuum pump capable of maintaining an isokinetic sampling rate while continuously withdrawing a portion of the stack gases through the sampling train. The pump intake vacuum is measured to within 13 mm Hg (0.5 in Hg) by a vacuum gauge attached to the vacuum line connecting the pump to the last impinger outlet. The sample flow rate is controlled by a combination of coarse and fine (by-pass) valves.

Metering System. A calibrated dry gas meter with inlet and outlet temperature sensors. The meter shall be calibrated according to the procedures specified in Method F, Reference Method EPS 1/RM/8. The temperature sensors must be capable of measuring to within 3°C (5°F).

Orifice. A calibrated orifice connected to the outlet of the dry gas meter. The orifice shall be calibrated according to the procedure specified in Method F, Reference Method EPS 1/RM/8.

Differential Pressure Indicators. The devices, such as inclined manometers, must be capable of measuring the pitot tube velocity pressure (Δp) and the pressure drop across the orifice to within 0.1 mm (0.005 in) H_2O on the 0 to 25 mm (0 to 1 in) H_2O scale, and 1 mm (0.05 in) H_2O on the 25 to 250 mm (1 to 10 in) H_2O scale. An inclined manometer filled with liquid of the corresponding density is considered a primary standard. Other devices must be calibrated against a primary standard prior to the test. A more sensitive device shall be required when Δp values are less than 1.3 mm H_2O (0.05 in H_2O).

Barometer. A barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg). The device must be calibrated against a primary standard prior to the test. Alternatively, the uncorrected atmospheric pressure provided by the local weather office may be used with an adjustment for the elevation above sea level of the sampling site. Deduct 2.5 mm Hg per 30.5 m (0.1 in Hg per 100 ft) elevation above sea level.

Timekeeping Device. A timekeeping device is required to measure the elapsed sampling time and the dwell time at each traverse point. The time keeping device must have a minimum resolution of 0.1 minutes.

3.2 Sample Recovery

The following items are required for sample recovery:

Probe and Cyclone Brushes. Nylon bristle brushes of a length and diameter suitable for cleaning the $PM_{2.5}$ cyclone and the probe. Teflon tubing brush handles are recommended for acetone and water rinsing. When not in use, the probe and cyclone brushes should be stored within a plastic bag, to avoid contamination.

Desiccator. An air tight laboratory cabinet to stabilize the temperature and moisture level of filters and evaporation residues prior to weighing. The desiccators must contain a tray with silica gel or equivalent desiccant, at ambient laboratory temperature.

Balances. An analytical balance capable of weighing the filter, $PM_{2.5}$ cyclone and probe rinse residues to within ± 0.1 mg or less, and a trip or top loading balance capable of weighing loaded impingers to within ± 0.1 g or less.

Drying Oven. A vented temperature-controlled oven capable of controlling the temperature to 105°C (221°F) to within 3°C (5°F).

Miscellaneous. Wash bottles, sufficient quantity of sample containers large enough to hold all rinses and Petri dishes for holding the filter samples. All items shall be made of materials such as glass, Teflon, or polypropylene which are chemically inert to both the samples and the reagents used for sample recovery.

4. Reagents and Materials

4.1 Sample Collection

The following items are required for sample collection:

Filter. A flash-fired glass fibre filter (organic binder removed) of a diameter compatible with the filter holder with an efficiency of at least 99.95% for 0.3 µm particles in accordance with the most recent version of ASTM Standard D2986. The manufacturer's test data for the filter is sufficient. The filter material must be chemically inert to stack gas components such as sulphur dioxide (SO_2). Depending on the nature of the source and the analyses required, other types of filter media may be used, subject to approval by Environment Canada.

The filter must be desiccated to a constant weight before being used. This may be accomplished by desiccating the filter for at least 24 hours with silica gel or equivalent desiccant at 20 ± 6°C (68 ± 10°F). Weigh the filter to the nearest 0.1 mg at intervals of six hours or more in a room where the relative humidity is 50% or less. The weighing must be completed within two minutes after the filter is removed from the desiccator. The constant weight is attained when the difference between two consecutive readings is less than 0.5 mg. Place the pre-weighed filter in a clean labeled Petri dish to prevent contamination during transportation to the sampling site.

Miscellaneous. Distilled or deionized water, crushed ice, fluoropolymer O-rings to seal joints and indicating type 6-16 mesh silica gel.

4.2 Sample Recovery

The following items are required for sample recovery:

Acetone. Reagent grade, low-residue acetone less than 0.001 percent by weight.

Water. The quality of the distilled or deionized water shall conform to specifications for Type II water given by the most recent version of Standard ASTM D1193.

Sample Jars. Chemically inert wide-mouth sample jars of a capacity of 250 mL to store the rinses of the nozzle, cyclone, probe liner and the front half of the filter holder.

Petri Dishes. Glass or plastic Petri dishes of a size greater than the filter.

The procedures in Section 5.1 of Method H are recommended for the weighing of rinse residues (use of reference jar).

5. Procedures

5.1 Sample Collection

Preliminary. In the absence of any previous knowledge of the stack variables, a preliminary test should be conducted to obtain the following data:

- Number and location of the traverse points in accordance to Method A, Reference Method EPS 1/RM/8.
- Velocity profile across the stack (Method B, Reference Method EPS 1/RM/8). The use of the pitot-$PM_{2.5}$ cyclone head assembly is recommended.
- Stack temperature and pressure (Method B, Reference Method EPS 1/RM/8).
- Stack gas molecular weight (Method C, Reference Method EPS 1/RM/8).
- Stack gas moisture content (Method D, Reference Method EPS 1/RM/8).

Since the nozzle on the cyclone is not situated at the extreme tip of the probe assembly, it may be impossible to locate the nozzle on the last traverse point on the far wall. In this case, place the nozzle at the second last point.

Sampling Rate Selection. The cutoff diameter of the $PM_{2.5}$ cyclone determines the sampling rate. At the same time, the isokinetic criteria must also be met at each traverse point. These simultaneous requirements may be met by varying dwell time at each point, changing nozzles and biasing the sampling up to 20% (up to 10% for particulate matter) with respect to the isokinetic rate.

Using the information from the preliminary stack run (P_{bar}, Δp_s, T_s, B_{wo}, $\%O_2$ and $\%CO_2$) determine M_s and M_d. Calculate the viscosity of the stack gas and the Cunningham factor. These will be used to determine the following parameters at each traverse point.

a) Nozzle diameter
b) Sampling rate or the pressure difference across the orifice (ΔH)
c) Dwell time

So as to meet the following conditions:

a) Dwell times proportional to stack gas velocity, with a target average dwell time of five minutes per reading
b) 2.25 microns ≤ cutoff diameter ≤ 2.75 microns
c) 80% ≤ isokineticity ≤ 120% (90% ≤ isokineticity ≤ 110% for PM)

Each of these conditions must be met in ≥ 90% of the traverse readings.

An example of a decision chart for selecting the $PM_{2.5}$ cutoff and sampling rate is illustrated in Figure G-9. Other iterative approaches are possible to meet the cutoff and the isokinetics. The desired sampling rate through the nozzle, Q_{nozzle}, is the product of the actual local velocity at the nozzle tip (U_s) and the area of the nozzle. The velocity is calculated using Equation B-2 of Method B, Reference Method EPS 1/RM/8. Once the iso-adjusted sampling rate through the nozzle is determined, the orifice setting, ΔH, is calculated using Equation F-3 of Method F, Reference Method EPS 1/RM/8.

Dwell Time Selection. Based on the preliminary traverse data calculate the average stack gas velocity, which will become the reference or "anchor" velocity for the dwell time calculations of all the points of the test run. The dwell time for each point is calculated at the time of actual sampling as follows:

$$\text{Dwell Time}_{point} = \text{Dwell Time}_{average} * U_s / U_{anchor}$$

For convenience, round the dwell time to either to the closest 15 seconds or tenth of a minute, depending on the units of the timekeeping device being used. Use the actual stack gas velocities measured during sampling to calculate the dwell time at each traverse point.

Total Test Run Time. After calculating the anchor velocity and determining the number of traverse points, choose an average dwell time and the number of readings per point that will result in a sample of at least 1.5 m^3 at dry reference conditions. The average dwell time for all the readings must not exceed five minutes and multiple readings may be required at each traverse point.

Sampling Train Preparation. Prepare the sample train in a clean area to minimize contamination. Mark the probe with heat resistant markings to denote the location of each sampling point. Use a pair of tweezers to place the labeled and tared filter in the filter holder.

Place 100 mL of water in each of the first and second impingers. Leave the third impinger empty. Place approximately 100 to 300 grams silica gel in the fourth impinger. Record the weight to the nearest 0.5 g of each impinger on the Moisture Analysis Data Sheet (Figure G-6).

Set up the sampling train as in Figure G-5. Adjust the filter compartment and probe heating system to maintain a temperature of 120 ± 14°C (248 ± 25°F) or such other temperature as is necessary to prevent blinding of the filter due to condensation.

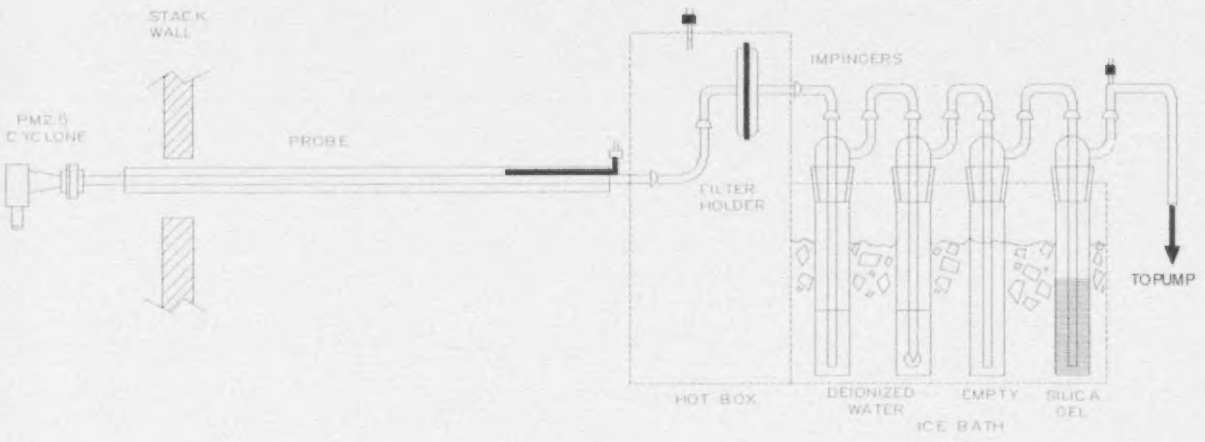


Figure G-5 Method G Schematic

Conduct a mandatory pre-test leak check of the sampling train by plugging the nozzle inlet and pulling a vacuum of 380 mm Hg (15 in. Hg) for at least one minute. The leakage rate must be less than 0.57 L/min (0.02 ft^3/min) or 4% of the estimated average sampling rate, whichever is less. Sampling must not proceed until the leakage rate is acceptable. Record the actual leakage rate on the Particulate Sampling Data Sheet (Figure G-10). Place ice and water in the impinger box before sampling.

It is recommended to conduct a pre-test leak check of the stack gas velocity measurement system as follows:
a) blow through the pitot impact opening until at least 7.6 cm (3.0 in) water velocity head registers on the manometer; then close off the impact opening. The pressure shall remain stable for at least 15 seconds
b) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3.0 in) water column reading.
Other equivalent leak-check procedures may be used.

Sampling Train Operation. Clean the portholes prior to sampling to avoid the extraneous pickup of settled dust in the nozzle. Insert the cyclone assembly through the port ensuring that the nozzle does not scrape against the port wall. Seal the port. To begin sampling, record the initial dry gas meter reading and point the selected nozzle directly into the gas stream at the first sampling point. Read the Δp and the stack gas temperature. Hold the probe at this position for a minimum of five minutes so that the temperature of the $PM_{2.5}$ cyclone reaches the stack gas temperature within ± 28°C (± 50°F).

Using a spreadsheet or a programmable calculator, determine the dwell time and the orifice setting that result in:

a) 2.25 microns ≤ $PM_{2.5}$ cyclone cutoff diameter ≤ 2.75 microns
b) 80% ≤ isokineticity ≤ 120% (90% ≤ isokineticity ≤ 110%, for PM)

Note: *If the preliminary traverse was performed just preceding sampling, the point-by-point nozzle diameters and orifice settings determined with the preliminary data may, in most cases, be utilized. Significant variation in stack temperature or velocity from the preliminary measurements will require these settings to be determined while sampling.*

Simultaneously start the vacuum pump and the timekeeping device. Rapidly adjust the flow control valves to the desired orifice setting. Sample all traverse points while maintaining the point-specific dwell time, the $PM_{2.5}$ cutoff diameter and the isokinetics. Add more ice and water to the ice box, as required, to maintain the temperature of the last impinger exit in the range of 0° to 20°C (32° to 68°F).

Record instrumentation readings on the Particulate Sampling Data Sheet (Figure G-10) at each traverse point. Readings must also be recorded before and after a leak check and when the sampling is halted. The total sample volume must be at least 1.5 m^3 measured at dry reference conditions. This target volume may require two or more consecutive readings per traverse point. Record the dwell times to the nearest 15 seconds or tenth of a minute to facilitate record keeping.

Sites where the point-to-point gas velocity varies by more than ± 20% (± 10% for PM) may require the use of two or more nozzles per test run to meet the isokinetic criteria. If the preliminary traverse or previous test run indicates that all the traverse points may be sampled using the same nozzle, then move the probe to the next point. If the preliminary traverse or previous test run indicates that more than one nozzle is necessary, then sample first the traverse points corresponding to the same nozzle. Then turn off the pump and immediately withdraw the probe from the stack. Replace the nozzle without delay and store the replaced nozzle in a clean sealed container. Do not leak test the train. Reinsert the probe and continue the test run.

When it is necessary to halt sampling temporarily, either to dismantle the sampling train during port changeover or to change a train component other than the nozzle, turn off the pump and immediately withdraw the probe from the stack. Separate the $PM_{2.5}$ cyclone, plug the end of the probe and conduct a mandatory leak check on the rest of the train by pulling a vacuum equal or greater than the maximum recorded during sampling. Record the actual leakage rate. If the leakage rate exceeds 0.57 L/min (0.02 ft^3/min) or 4% of the sampling flow rate, the test is invalid. If the leakage rate is acceptable, proceed with dismantling the sampling train or changing the train component. Before continuing with the test, conduct a mandatory leak check (without the $PM_{2.5}$ cyclone) by following the pre-test leak check procedures used during sampling train preparation. Then, install and tighten the $PM_{2.5}$ cyclone connection.

When the test is completed, carefully remove the $PM_{2.5}$ cyclone, plug the end of the probe and conduct a mandatory post-leak check on the sampling train by pulling a vacuum equal to or greater than the maximum value observed during sampling. Record the actual leakage rate, which must be less than 0.57 L/min (0.02 ft^3/min) or 4% of the sampling rate, whichever is less. If the leakage rate is acceptable proceed to recover the samples.

Conduct a mandatory post-test leak check of the stack gas velocity measurement system following the same procedure as the recommended pre-test leak check.

5.2 Sample Recovery

Carefully disconnect the probe from the sampling train and seal all openings. Seal the openings of the $PM_{2.5}$ cyclone. Exercise care in moving the train components from the test site to the sample recovery area to minimize the loss of collected particulate matter or contamination.

Weigh each of the components that collected condensate or moisture to the nearest 0.5 g and record the results in the Moisture Analysis Data Sheet (Figure G-6). If chemical analysis of the condensate is required, then transfer the condensate and corresponding rinses into an additional container. Seal and label the container and mark the liquid level. Discard or recycle the spent silica gel. Use a fine tip marker to mark the levels on all the containers.

Partition the train samples as follows:

$PM_{2.5}$ Cyclone Rinse (Containers No. 1 and 2). Carefully wipe any visible traces of particulate from the exterior surfaces of the nozzle and cyclone. Wash and brush the interior surfaces of the nozzle and the cyclone with approximately 100 mL water. Store these rinses in Container No.1. This container does not need to be tared. Then wash and brush the interior surfaces of the nozzle and the cyclone with approximately 100 mL of acetone. Place these washings into Container No. 2. This container needs to be tared or quantitatively transferred to a tared container for gravimetric determination. Seal and label the containers and mark the liquid levels.

Cyclone Exit Tube, Probe Liner and Front Half of Filter Holder Rinse (Container No. 3 and 4). Wash and brush the interior surfaces with approximately 100 mL of water. Store these rinses in Container 3. This container does not need to be tared. Then wash and brush the interior surfaces with approximately 100 mL of acetone. Place these washings into Container No. 4. This container needs to be tared or quantitatively transferred to a tared container for gravimetric determination. Seal and label the containers and mark the liquid levels.

Filter (Container No. 5). Use a pair of clean tweezers or a sharp edge to transfer the glass fibre filter and any loose material that may adhere to the filter support into a Petri dish. Seal and label the sample container.

Blank (Containers No. 6 and 7). Place 100 mL of water and 100 mL of acetone, each one taken directly from the corresponding wash bottle being used, into Containers No. 6 and 7, respectively. Container No. 6 does not need to be tared. Container No. 7 needs to be tared or quantitatively transferred to a tared container upon receipt for gravimetric determination. Seal and label the containers and mark the liquid levels.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Weighing Conducted by: _____

Component	Content	Weight (g)
First Impinger	100 mL deionized or distilled water	Final _____ Initial _____ Gain _____ (a)
Second Impinger	100 mL deionized or distilled water	Final _____ Initial _____ Gain _____ (b)
Third Impinger	Empty	Final _____ Initial _____ Gain _____ (c)
Fourth Impinger	~ 200 g silica gel	Final _____ Initial _____ Gain _____ (d)
Total Moisture gain = a + b + c + d		_____ g

Figure G-6 Moisture Analysis Data Sheet

5.3 Sample Analysis

All analyses shall be performed in a clean laboratory equipped with a fume hood. The relative humidity of the room in which weighing is performed should be maintained at or below 50%.

Containers No. 1 and 2 ($PM_{2.5}$ Cyclone Water and Acetone Rinse). Note the liquid level of the containers and determine if leakage occurred during transport. If there is a loss of the sample, the +$PM_{2.5}$ results will be invalid. Allow the contents of Container No. 2 containing the acetone to evaporate to dryness at room temperature in a fume hood. Quantitatively transfer the contents of Container No. 1 to the residue of Container No. 2. Place the open container in a 105°C (220°F) oven to evaporate the aqueous phase. Then place the open container in a desiccator for 24 hours. Weigh until consecutive readings are less than 0.5 mg apart and record the result to the nearest 0.1 mg on the Particulate Analytical Data Sheet (Figure G-7). A minimum of six hours must be allowed between consecutive readings.

Containers No. 3 and 4 (Water and Acetone Rinses of Cyclone Exit Tube, Probe Liner and Front Half of Filter Holder). Note the liquid level of the container and determine if leakage occurred during transport. If there is a loss of the sample, the filterable $PM_{2.5}$ result will be invalid. Allow the contents of Container No. 4 containing the acetone to evaporate to dryness at room temperature in a fume hood. Quantitatively transfer the contents of Container No. 3 to the residue of Container No. 4. Place the open container in a 105°C (220°F) oven to evaporate the aqueous phase. Then place the open container in a desiccator for 24 hours. Weigh until consecutive readings are less than 0.5 mg apart and record the result to the nearest 0.1 mg on the Particulate Analytical Data Sheet (Figure G-7). A minimum of six hours must be allowed between consecutive readings.

Container No. 5 (Filter). Transfer the filter and any loose particulate matter and filter material from the Petri dish to a tared weighing dish. Place the open container in a desiccator for 24 hours. Weigh until consecutive readings are less than 0.5 mg apart and record the result to the nearest 0.1 mg on the Particulate Analytical Data Sheet (Figure G-7). A minimum of six hours must be allowed between consecutive readings.

Containers No. 6 and 7 (Water and Acetone Blanks). Note the liquid level of the container and determine if leakage occurred during transport. If there is a loss of the sample, then the blank correction will be invalid. Allow the contents of Container No. 7 containing the acetone to evaporate to dryness at room temperature in a fume hood. Quantitatively transfer the contents on Container No. 6 to the residue of Container No. 7. Place the open container in a 105°C (220°F) oven to evaporate the aqueous phase. Then place the open container in a desiccator for 24 hours. Weigh until consecutive readings are less than 0.5 mg apart and record the result to the nearest 0.1 mg on the Particulate Analytical Data Sheet (Figure G-7). A minimum of six hours must be allowed between consecutive readings.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Test Conducted by: _____

Condition of Samples as Received			
Container/ Jar No.		**Description**	**Observations**
1		Water and acetone rinses of $PM_{2.5}$ nozzle and cyclone	Leakage during transport? Note condition of seal
2			Leakage during transport? Note condition of seal
3		Water and acetone rinses of cyclone tube exit, probe liner and front-half of filter holder	Leakage during transport? Note condition of seal
4			Leakage during transport? Note condition of seal
5		Filter	Note condition
6		Water/Acetone blank	Leakage during transport? Note condition of seal
7			Leakage during transport? Note condition of seal

Train Component	Description	Final Weight (mg)	Tare Weight (mg)	Weight of Particulate (mg)
Container 2	+$PM_{2.5}$			(a)
Container 4 Container 5	$PM_{2.5}$ Filter			(b) (c)
Container 7	Water/Acetone blank			(d)

Filterable $PM_{2.5}$	=	$W_{PM2.5} = (b - d) + c$ $W_{PM2.5} =$ _____ mg
Total (filterable) Particulate Matter	=	$W_{PM} = a + (b - d) + c$ $W_{PM} =$ _____ mg

Notes:

- *The weight of the water/acetone blank must not exceed 2 mg*
- *Do not correct for negative blank values*
- *No blank corrections are allowed when blank residue is above 2 mg*
- *The value for W_{PM} is valid only when the isokinetics are between 90 to 110%*

Figure G-7 Particulate Analytical Data Sheet

6. Calculations (SI Unit Equations)

See Appendix G for Imperial Unit Equations

During a test run, field data should be entered in the units of the stack sampling equipment (Imperial or common practice metric units). The equations of this method follow the common practice units, to facilitate its application and to avoid errors. The results may be converted, if necessary, to the units specified in the applicable standards or guidelines. The use of term weight actually refers to mass. The abbreviation "W" is used for weight (or mass) to avoid confusion with "M" which is used for molecular weight (or mass).

The method detection limit for the gravimetric determination of residues from jars is 0.42 mg. The total expanded uncertainty (95% probability) for filterable PM (+$PM_{2.5}$ and $PM_{2.5}$) is 1.0 mg. Based on 15 co-located paired trains at a Portland cement plant, the relative deviation is 2.9% and 3.1% for filterable $PM_{2.5}$ and filterable particulate matter, respectively.

Stack Gas Viscosity. The viscosity of the stack gas μ_s is calculated using Equation G-1.

$$\mu_s = C_1 + C_2\sqrt{T_s} + C_3 T_s^{-2} + C_4 \%O_{2,w} - C_5 B_{wo} + C_6 B_{wo} T_s^2 \qquad \textbf{Equation G-1}$$

where:
C_1 = -150.3162 (micropoise)
C_2 = 18.0614 (micropoise/$K^{0.5}$)
C_3 = 1.19183 x 10^6 (micropoise/K^2)
C_4 = 0.591123 (micropoise)
C_5 = 91.9723 (micropoise)
C_6 = 4.91705 x 10^{-5} (micropoise/K^2)

As the oxygen level in a stack gas is usually measured on a dry basis, the following relationship must be used to calculate the percent of oxygen in the stack gas on a wet basis.

$\%O_{2,w} = (1-B_{wo}) \times \%O_{2,d}$

Cunningham Correction Factor. The Cunningham correction factor is calculated using Equation G-2. Set D_{50} at 2.5.

$$C = 1 + (2.5985 \times 10^{-2})\left(\frac{\mu_s}{P_s D_{50}}\right)\left(\frac{T_s}{M_s}\right)^{0.5} \qquad \textbf{Equation G-2}$$

Cyclone Reynolds Number. The Reynolds number at sampling conditions is calculated using Equation G-3.

$$\mathrm{Re} = 5005.65\left[\frac{P_s M_s Q_{nozzle}}{\mu_s T_s}\right] \qquad \textbf{Equation G-3}$$

Q_{nozzle} is the product of the actual velocity at the nozzle for each traverse point, the area of the nozzle and an iso-adjustment factor to meet the D_{50} cutoff.

Cyclone cutoff. The cutoff of the $PM_{2.5}$ cyclone, for a given sample flow rate and Reynolds number is calculated using Equation G-4 or G-5.

For a Reynolds number less than 3162 use Equation G-4.

$$D_{50} = 0.4273 \left(\frac{\mu_s}{Q_{nozzle}} \right)^{1.1791} \left(\frac{1}{C} \right)^{0.5} \left(\frac{T_s}{P_s M_s} \right)^{0.6790}$$ **Equation G-4**

For a Reynolds number greater than 3162 use Equation G-5.

$$D_{50} = 0.5071 \left(\frac{\mu_s}{Q_{nozzle}} \right)^{0.8058} \left(\frac{1}{C} \right)^{0.5} \left(\frac{T_s}{P_s M_s} \right)^{0.3058}$$ **Equation G-5**

Volume of Stack Gas Sample. Correct the total sample volume measured by the gas meter to reference temperature and pressure conditions (298 K and 101.325 kPa) using equation G-6.

$$(V_m)_{ref} = \gamma V_m \frac{T_{ref} (P_{bar} + \Delta H_{avg})}{(T_m)_{avg} P_{ref}}$$ **Equation G-6**

Volume of Water Vapour. Calculate the volume of water vapour in the stack gas sample at reference conditions using Equation G-7.

$$(V_w)_{ref} = 0.00136 W_{H_2O}$$ **Equation G-7**

Moisture Content of the Stack Gas. Calculate the volumetric fraction of water vapour in the stack gas at reference conditions using equation G-8.

$$B_{wo} = \frac{(V_w)_{ref}}{(V_w)_{ref} + (V_m)_{ref}}$$ **Equation G-8**

For saturated or supersaturated stack gas, use a psychrometric chart or equation to determine B_{wo}.

Absolute Stack gas Pressure. Calculate the absolute stack gas pressure using Equation G-9.

$$P_s = P_{bar} + \Delta p_s$$ **Equation G-9**

Stack Gas Molecular Weight. Calculate the stack gas molecular weight on a wet basis using Equation G-10.

$$M_s = M_d (1 - B_{wo}) + 18 B_{wo}$$ **Equation G-10**

Note: Use Equation C-2 or C-3 of Method C to calculate M_d.

Stack Gas Velocity. Calculate the stack gas velocity measured at each traverse point using Equation G-11.

$$U_S = 128.95 C_p \left(\frac{\Delta p T_s}{P_s M_s} \right)^{1/2}$$ **Equation G-11**

Volumetric Stack Gas Flow Rate. Calculate the volumetric flow rate of the stack gas on a dry basis and reference conditions using Equation G-12.

$$Q_s = 3600(U_s)_{avg} A_s (1 - B_{wo}) \frac{T_{ref} P_s}{(T_s)_{avg} P_{ref}} C_{BF}$$ **Equation G-12**

Adjust the stack cross-section area by the average area blocked by the $PM_{2.5}$ sizing head and probe assembly during the run. For cylindrical stacks and probes of standard diameter (2.5 cm, 1 inch) adjust the stack cross-section by the factor shown in Figure G-8.

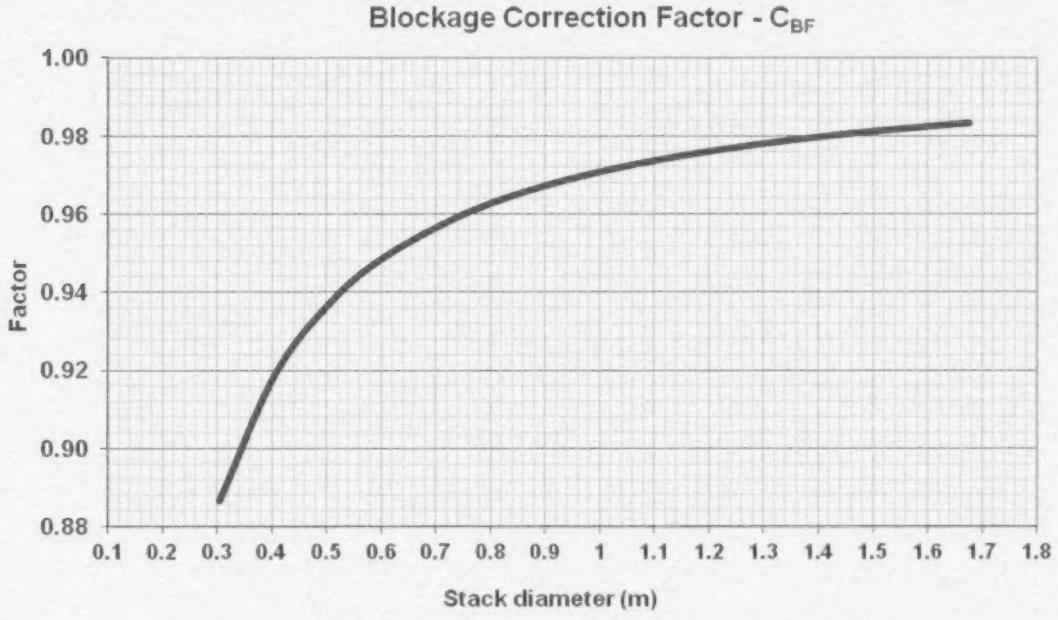


Figure G-8 Blockage Correction Factor (C_{BF})

Concentration of $PM_{2.5}$. Determine the concentration of $PM_{2.5}$ in the stack gas at reference conditions using Equation G-13. $W_{PM2.5}$ is the sum of the residues from the cyclone exit tube, probe, front-half rinse and the filter corrected for the blank.

$$C_{PM\,2.5} = \frac{W_{PM\,2.5}}{(V_m)_{ref}}$$ **Equation G-13**

Concentration of PM. Determine the concentration of PM in the stack gas at reference conditions using Equation G-14. W_{PM} is the sum of the residues from all the fractions in the front half.

$$C_{PM} = \frac{W_{PM}}{(V_m)_{ref}}$$ **Equation G-14**

Mass Emission Rate. Determine the mass emission rate of $PM_{2.5}$ or PM using Equation G-15.

$$ER_{PM\,2.5}\ or\ ER_{PM} = 10^{-6}\,(C_{PM\,2.5}\ or\ C_{PM})\,Q_s$$ **Equation G-15**

Isokineticity. Verify the isokineticity for each traverse point using Equation G-16.

$$I = \frac{\gamma \dfrac{(V_m)_j}{t} \dfrac{1}{(1-B_{wo})} (P_{bar} + \Delta H) T_s}{6 \times 10^{-5} \left(\dfrac{\pi}{4}\right) (N_d)^2 (T_m)_{avg} P_s U_s} \times 100$$

Equation G-16

A test run for filterable $PM_{2.5}$ shall be considered valid with respect to isokineticity and cut size providing that:

a) 90% or more of the isokineticity values, I, calculated for all readings are within the range of 80% to 120%, i.e. 80% ≤ I ≤ 120%.
b) The arithmetic average of all the isokinetic values is within the range of 80 to 120%, i.e. 80% ≤ I ≤ 120%.
c) 90% or more of the cyclone cutoff diameters calculated for all readings are within the range of 2.25 micron to 2.75 micron i.e. 2.25 micron ≤ cutoff diameter ≤ 2.75 micron
d) The arithmetic average of all the cyclone cutoff diameters is within the range of 2.25 micron to 2.75 micron i.e. 2.25 micron ≤ cutoff diameter ≤ 2.75 micron

A test run for PM shall be considered valid with respect to isokineticity providing that:

e) 90% of more of the isokineticity values, I, calculated for all readings are within the range of 90 to 110%, i.e. 90% ≤ I ≤ 110%.
f) The arithmetic average of all the isokinetic values is within the range of 90 to 110%, i.e. 90% ≤ I ≤ 110%.

7. Nomenclature

A_s – inside cross sectional area of stack or duct, m^2

B_{wo} – proportion by volume of water in the stack gas, dimensionless

C_{BF} – average blockage correction factor, dimensionless

C_p – pitot tube coefficient, dimensionless

$C_{PM2.5}$ – concentration of $PM_{2.5}$ in the stack gas on a dry basis at reference temperature and pressure conditions, mg/m^3

C_{PM} – concentration of filterable particulate matter in the stack gas on a dry basis at reference temperature and pressure conditions, mg/m^3

D_{50} . $PM_{2.5}$ cyclone cutoff diameter (microns) at the sampling conditions

$ER_{PM2.5}$ – emission rate of filterable $PM_{2.5}$ particulate matter, kg/h

ER_{PM} – emission rate of filterable particulate matter, kg/h

ΔH – pressure drop across orifice meter for each traverse point, kPa

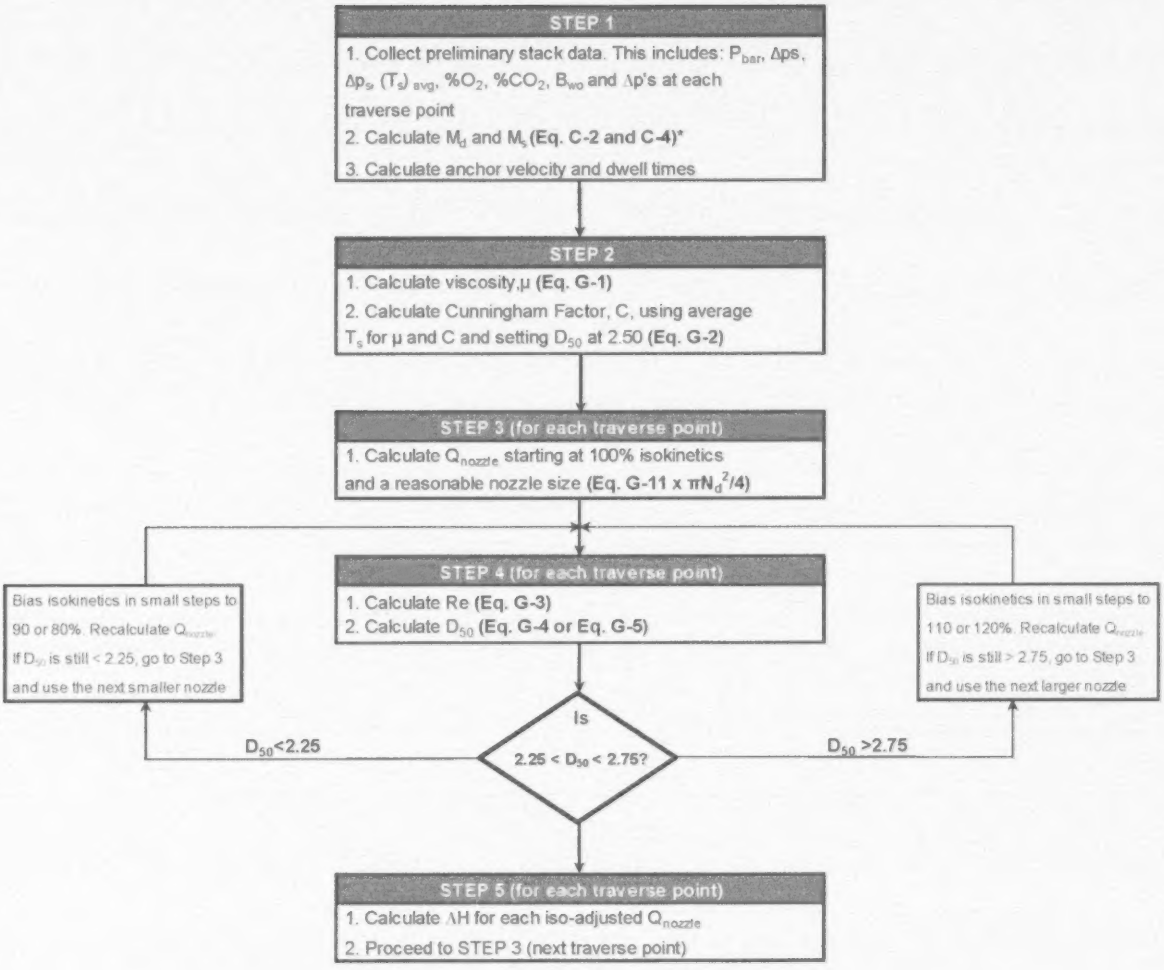
I – isokineticity, i.e., the ratio of the sampling velocity through the nozzle to the velocity of the undisturbed gas stream at each traverse point, %

j – the j^{th} traverse point, dimensionless

M_s – molecular weight of stack gas on a wet basis, kg/kmol

M_d	– molecular weight of stack gas on a dry basis, kg/kmol
N_d	– inside diameter of the sampling nozzle, mm
$\%O_{2,d}$	– oxygen level in the stack gas on a dry basis, %(v/v)
$\%O_{2,w}$	– oxygen level in the stack gas on a wet basis, %(v/v)
Δp	– stack gas velocity pressure at each traverse point, kPa
Δp_s	– stack static pressure, kPa
P_{bar}	– barometric pressure at sampling site, kPa
P_{ref}	– reference pressure, 101.325 kPa
P_s	– absolute stack gas pressure, kPa
Q_{nozzle}	– iso-adjusted volumetric gas flow rate through the nozzle at the inlet conditions, L/m
Q_s	– volumetric stack gas flow rate on a dry basis at reference temperature and pressure conditions, m^3/h
t	– total run time per reading, min
$(T_m)_{avg}$	– arithmetic average of the inlet and outlet dry gas meter temperatures at each traverse point, K
T_{ref}	– reference temperature, 298 K
T_s	– stack gas temperature at each traverse point, K
$(T_s)_{avg}$	– arithmetic average of the stack gas temperatures, K
U_s	– stack gas velocity at each traverse point, m/s
$(U_s)_{avg}$	– arithmetic average of the stack gas velocities, m/s
V_m	– volume of stack gas sample at dry gas meter conditions, m^3
$(V_m)_j$	– volume of stack gas sample at dry gas meter conditions for the j^{th} traverse point, m^3
$(V_m)_{ref}$	– volume of stack gas sample at reference temperature and pressure conditions, m^3
$(V_w)_{ref}$	– volume of water vapour at reference conditions, m^3
W_{H2O}	– weight of water condensed in the impingers, g
$W_{PM2.5}$	– weight of filterable $PM_{2.5}$ collected during the test run, mg
W_{PM}	– weight of filterable particulate collected during the test run, mg
μ_s	– stack gas viscosity, micropoise
π	– pi, 3.14159
γ	– dry gas meter correction factor, dimensionless
0.00136	– m^3 water vapour/g water

18	– molecular weight of water, kg/kmol
128.95	– dimensional constant, $(m/s)[(kg/kmol)/K]^{1/2}$
3600	– conversion factor, seconds per hour, s/h
10^{-6}	– conversion, kg/mg
6×10^{-5}	– conversion factor, $(m^2/mm^2)(s/min)$



NOTES

1. More than one nozzle may be available to meet a D_{50} at a certain velocity. Choose the sizes that result in the least amount of nozzles for a run.

2. It is recommended to use a more stringent isokinetic criteria such as 93% <%ISO <107% for PM and 85% <%ISO <115% for $PM_{2.5}$

** Method C, Reference Method EPS 1/RM/8*

Figure G-9 Decision Chart for Setting Sampling Rate for D_{50}

Page _____of _____

ISOKINETIC SAMPLING DATA SHEET – $PM_{2.5}$

Plant & Location		Run No.		Date		Start Time	
Sampling Location (Source)		Method		Operator(s)		Finish Time	

Stack Diameter	m	Cyclonic Flow Checked ?		Stack/Duct Schematic	Bar Pressure (P_b) kPa
Primary Filter type		Y	N		Static Pressure ($\triangle p_s$) kPa
Secondary Filter type		Average angle °			Pitot Coefficient (C_p)
Liner material		**Process Stable?**			Meter Coefficient (γ)
Nozzle No.	Diam. mm				Orifice Coefficient (K_o)
Nozzle No.	Diam. mm	**Capacity ___%**			or DH@ kPa
Nozzle No.	Diam. mm				Moisture (B_{wo})
Initial Leak check	L/min @	380 mm Hg vacuum			CO_2 (%)
Post Leak check	L/min @	mm Hg vacuum			O_2 (%)

Traverse

Point No.	Nozzle No.	Dwell Time (min)	Dry Gas Meter Reading (L)	Velocity Pressure $\triangle p$ (kPa)	Orifice Pressure $\triangle H$ (kPa)	Temperatures (°C)							Pump Vacuum (mm Hg)
						Stack Gas	Probe	Primary Filter	Secondary Filter	Last Impinger	DGM Inlet	DGM Outlet	

Figure G-10 Particulate Sampling Data Sheet

APPENDIX G. Equations with Imperial Units

Stack Gas Viscosity: The viscosity of the stack gas μ_s is calculated using Equation G-1.

$$\mu_s = C_1 + C_2\sqrt{T_s} + C_3 T_s^{-2} + C_4 \%O_{2,w} - C_5 B_{wo} + C_6 B_{wo} T_s^2 \qquad \textbf{Equation G-1}$$

where:
C_1 = -150.3162 (micropoise)
C_2 = 13.4622 (micropoise/$R^{0.5}$)
C_3 = 3.86153 x 10^6 (micropoise/R^2)
C_4 = 0.591123 (micropoise)
C_5 = 91.9723 (micropoise)
C_6 = 1.51761 x 10^{-5} (micropoise/R^2)

As the oxygen level in a stack gas is usually measured on a dry basis, the following relationship must be used to calculate the percent of oxygen in the stack gas on a wet basis.

$\%O_{2,w} = (1-B_{wo}) \times \%O_{2,d}$

Cunningham Correction Factor. The Cunningham correction factor is calculated using Equation G-2. Set D_{50} at 2.5.

$$C = 1 + (5.7193 \times 10^{-3})\left(\frac{\mu_s}{P_s D_{50}}\right)\left(\frac{T_s}{M_s}\right)^{0.5} \qquad \textbf{Equation G-2}$$

Cyclone Reynolds Number. The Reynolds number at sampling conditions is calculated using Equation G-3.

$$\mathrm{Re} = 8.640 \times 10^5 \frac{P_s M_s Q_{nozzle}}{\mu_s T_s} \qquad \textbf{Equation G-3}$$

Q_{nozzle} is the product of the actual velocity at the nozzle for each traverse point, the area of the nozzle and an iso-adjustment factor to meet the D_{50} cutoff.

Cyclone cutoff. The cutoff of the $PM_{2.5}$ sizer for a given sample flow rate and Reynolds number is calculated using Equation G-4 or G-5.

For a Reynolds number less than 3162 use Equation G-4.

$$D_{50} = 2.4302 \times 10^{-3}\left(\frac{\mu_s}{Q_{nozzle}}\right)^{1.1791}\left(\frac{1}{C}\right)^{0.5}\left(\frac{T_s}{P_s M_s}\right)^{0.6790} \qquad \textbf{Equation G-4}$$

For a Reynolds number greater than 3162 use Equation G-5.

$$D_{50} = 1.9723 \times 10^{-2}\left(\frac{\mu_s}{Q_{nozzle}}\right)^{0.8058}\left(\frac{1}{C}\right)^{0.5}\left(\frac{T_s}{P_s M_s}\right)^{0.3058} \qquad \textbf{Equation G-5}$$

Volume of Stack Gas Sample. Correct the total sample volume measured by the gas meter to reference temperature and pressure conditions (77°F and 29.92 in Hg) using equation G-7.

$$(V_m)_{ref} = \gamma V_m \frac{T_{ref}\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{(T_m)_{avg} P_{ref}}$$ **Equation G-6**

Volume of Water Vapor. Calculate the volume of water vapor in the stack gas sample at reference conditions using Equation G-7.

$$(V_w)_{ref} = 0.048 W_{H_2O}$$ **Equation G-7**

Moisture Content of the Stack Gas. Calculate the volumetric fraction of water vapor in the stack gas at reference conditions using equation G-8.

$$B_{wo} = \frac{(V_w)_{ref}}{(V_w)_{ref} + (V_m)_{ref}}$$ **Equation G-8**

For saturated or supersaturated stack gas, use a psychometric chart to determine B_{wo}.

Absolute Stack Gas Pressure. Calculate the absolute stack gas pressure using Equation G-9.

$$P_s = P_{bar} + \frac{\Delta p_s}{13.6}$$ **Equation G-9**

Stack Gas Molecular Weight. Calculate the stack gas molecular weight on a wet basis using Equation G-10.

$$M_s = M_d(1 - B_{wo}) + 18B_{wo}$$ **Equation G-10**

Note: Use Equation C-2 or C-3 of Method C to calculate M_d.

Stack Gas Velocity. Calculate the stack gas velocity measured at each traverse point using Equation G-11.

$$U_S = 85.52 C_p \left(\frac{\Delta p T_s}{P_s M_s}\right)^{1/2}$$ **Equation G-11**

Volumetric Stack Gas Flow Rate. Calculate the volumetric flow rate of the stack gas on dry basis and reference conditions using Equation G-12.

$$Q_s = 3600(U_s) A_s (1 - B_{wo}) \left(\frac{T_{ref} P_s}{(T_s)_{avg} P_{ref}}\right) C_{BF}$$ **Equation G-12**

Adjust the stack cross section area by the average area blocked by the $PM_{2.5}$ sizing head and probe assembly during the run. For cylindrical stacks and probes of standard diameter (2.5 cm, 1 in) adjust the stack cross section by the factor shown in Figure G-11.

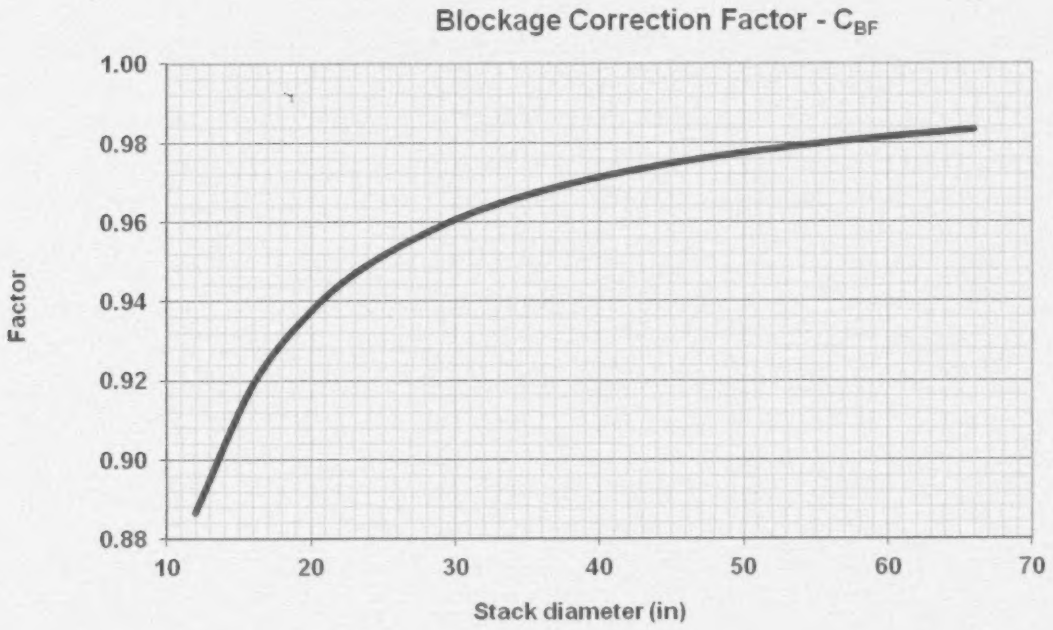


Figure G-11 Blockage Correction Factor (C_{BF}, Imperial)

Concentration of $PM_{2.5}$. Determine the concentration of $PM_{2.5}$ in the stack gas at reference conditions using Equation G-13. $W_{PM2.5}$ is the sum of the residues from the cyclone exit tube, probe, front-half rinse and the filter corrected for the blank.

$$C_{PM2.5} = \frac{W_{PM2.5}}{(V_m)_{ref}}$$ **Equation G-13**

Concentration of PM. Determine the concentration of PM in the stack gas at reference conditions using Equation G-14. W_{PM} is the sum of the residues from all the fractions in the front half.

$$C_{PM} = \frac{W_{PM}}{(V_m)_{ref}}$$ **Equation G-14**

Mass Emission Rate. Determine the mass emission rate of $PM_{2.5}$ or PM using Equation G-15.

$$ER_{PM2.5} \text{ or } ER_{PM} = 10^{-6}(C_{PM2.5} \text{ or } C_{PM})Q_s$$ **Equation G-15**

Isokineticity. Verify the isokineticity for each traverse point using Equation G-16.

$$I = \frac{\gamma \dfrac{(V_m)_j}{t} \dfrac{1}{(1-B_{wo})} \left(P_{bar} + \dfrac{\Delta H}{13.6}\right) T_s}{0.4167 \left(\dfrac{\pi}{4}\right) (N_d)^2 (T_m)_{avg} P_s U_s} \times 100$$

Equation G-16

Nomenclature

A_s	– inside cross sectional area of stack or duct, ft^2
B_{wo}	– proportion by volume of water in the stack gas, dimensionless
C_{BF}	– average blockage correction factor, dimensionless
C_p	– pitot tube coefficient, dimensionless
$C_{PM2.5}$	– concentration of $PM_{2.5}$ in the stack gas on a dry basis at reference temperature and pressure conditions, mg/ft^3
C_{PM}	– concentration of filterable particulate matter in the stack gas on a dry basis at reference temperature and pressure conditions, mg/ft^3
D_{50}	. $PM_{2.5}$ cyclone cutoff diameter (microns) at the sampling conditions
$ER_{PM2.5}$	– emission rate of filterable $PM_{2.5}$ particulate matter, kg/h
ER_{PM}	– emission rate of filterable particulate matter, kg/h
ΔH	– pressure drop across orifice meter for each traverse point, in of H_2O
I	– isokineticity, i.e., the ratio of the sampling velocity through the nozzle to the velocity of the undisturbed gas stream at each traverse point, %
j	– the j^{th} traverse point, dimensionless
M_s	– molecular weight of stack gas on a wet basis, lb/lb-mol
M_d	–molecular weight of stack gas on a dry basis, lb/lb-mol
N_d	– inside diameter of the sampling nozzle, in
$\%O_{2,d}$	– oxygen level in the stack gas on a dry basis, %(v/v)
$\%O_{2,w}$	- oxygen level in the stack gas on a wet basis, %(v/v)
Δp	– stack gas velocity pressure at each traverse point, in of H_2O
Δp_s	– stack static pressure at each traverse point, in of H_2O
P_{bar}	– barometric pressure at sampling site, in of Hg
P_{ref}	– reference pressure, 29.92 in of Hg
P_s	– absolute stack gas pressure, in of Hg

Q_{nozzle}	– iso-adjusted volumetric gas flow rate through the nozzle at the inlet conditions, ft^3/min
Q_s	– volumetric stack gas flow rate on a dry basis at reference temperature and pressure conditions, ft^3/hr
t	– total run time per reading, min
$(T_m)_{avg}$	– arithmetic average of the inlet and outlet dry gas meter temperatures at each traverse point, R
T_{ref}	– reference temperature, 537 R
T_s	– stack gas temperature at each traverse point, R
$(T_s)_{avg}$	– arithmetic average of the stack gas temperatures, R
U_s	– stack gas velocity at each traverse point, ft/s
$(U_s)_{avg}$	– arithmetic average of the stack gas velocities, ft/s
V_m	– volume of stack gas sample at dry gas meter conditions, ft^3
$(V_m)_j$	– volume of stack gas sample at dry gas meter conditions for the j^{th} traverse point, ft^3
$(V_m)_{ref}$	– volume of stack gas sample at reference temperature and pressure conditions, ft^3
$(V_w)_{ref}$	– volume of water vapour at reference conditions, ft^3
W_{H2O}	– weight of water condensed in the impingers, g
$W_{PM2.5}$	– weight of filterable $PM_{2.5}$ collected during the test run, mg
W_{PM}	– weight of filterable particulate collected during the test run, mg
μ_s	– stack gas viscosity, micropoise
π	– pi, 3.14159
γ	– dry gas meter correction factor, dimensionless
13.6	– conversion factor, in of H_2O/in of Hg
0.048	– ft^3 water vapour/g water
18	– molecular weight of water, 18 lb/lb-mol
85.52	– dimensional constant, (ft/s)[(in of Hg x lb/lb-mol)/(in of H_2O x R)]$^{1/2}$
3600	– conversion factor, seconds/hour
0.4167	– conversion factor, $(s/min)(ft^2/in^2)$
10^{-6}	– conversion, kg/mg

Method H: Determination of Condensable Particulate Matter (CPM)

1. Applicability

This method is used to measure condensable particulate matter (CPM) released from stationary sources. CPM is matter that is vapour phase at stack conditions but immediately forms a liquid or solid in the cooler ambient air once the stack gases are discharged to atmosphere. CPM is material that after passing through a filter held at 120 ± 14°C (248 ± 25°F) condenses at the near ambient temperature of this method. CPM is considered to be $PM_{2.5}$.

This method is used in conjunction with the Methods of Report EPS 1/RM/8, December 1993, *Standard Reference Method for Source Testing: Measurement of Releases of Particulate from Stationary Sources* and it is subject to their limitations regarding stack gas conditions.

2. Principle

Particulate matter is withdrawn from the stack and filtered at a temperature of 120 ± 14°C (248 ± 25°F). The filtered gas sample is then cooled in an ice-water jacketed coil condenser and any moisture that may condense is collected in a condensate trap or stemless impinger. Ethanol is added to this impinger to inhibit the oxidation of SO_2 that may be dissolved in the condensate. Any aerosol formed by the cooling of the sample gas is collected by a secondary or mist filter held at ambient temperature. At the completion of the test the sample is recovered quantitatively and the condensate is extracted with dichloromethane (DCM). The organic fraction is evaporated at ambient temperature, whereas the aqueous fraction is evaporated to near-dryness in a 105°C (220°F) oven. Both dryings are completed in a desiccator and the residue weighed.

Sampling isokinetically means that the velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sampling point.

Three valid tests are required for the determination of condensable particulate matter. Each test must be a minimum of two hours and collect at least 1.7 m^3 (60 ft^3) of stack gas on a dry basis at reference conditions. This method has not been evaluated beyond sampling durations of four hours and condensate catches beyond 250 grams. Contact Environment Canada if either of these conditions apply.

3. Apparatus

3.1 Sample Collection

The items required for sample collection are similar to that of Method G, with the following additions:

Condenser. Glass, coil type with water cooling jacket and ball joint endings, capable of cooling the sample stream to 30°C or less, as measured by the sensor installed in the secondary filter holder. Either horizontal or vertical condensers are acceptable. The water from the ice bath is used to cool the condenser.

Water pump. A pump to circulate ice water through the water jacket condenser.

Condensate Trap. A Greenburg Smith impinger without a stem or a glass condensate trap is required to collect the water knocked out in the upstream condenser.

Impingers. Three Greenburg-Smith impingers connected in series. The second and third impingers are modified by replacing the tips and impaction plates of the standard design with a 13 mm (0.5 in) ID glass tube extending to

within 13 mm (0.5 in) of the bottom of the impinger. The first impinger has the standard tip and impaction plate. The impingers are contained in an ice bath during sampling. A temperature sensor capable of measuring to within 1°C (2°F) shall be placed at the outlet of the last impinger.

Secondary (Mist) Filter Holder. A glass filter holder fitted with a Teflon support capable of holding a greater than 47 mm membrane filter, with an inert filter support (Teflon plate, stainless steel perforated plate) fitted with soft sealing surfaces to prevent gas leakage into or around the filter. A Teflon filter support with fluoropolymer O-rings is recommended. The back half of the secondary filter holder must be fitted with a thermocouple to measure the compartment temperature to within 1°C (2°F).

The secondary filter holder is located above the impingers. The secondary filter holder may require heating to prevent freezing and plugging if the test is conducted at sub-zero ambient temperatures. In this situation, however, the temperature of the secondary filter holder must not exceed 30°C.

Pipet. Graduated to measure one to five mL of liquid.

3.2 Sample Recovery

The items required for sample collection are similar to that of Method G.

4. Reagents and Materials

4.1 Sample Collection

Water. HPLC grade, for rinsing and sample recovery, shall have an evaporation residue ≤ 0.001 percent by weight.

Ethanol. Reagent grade, for inhibiting the oxidation of SO_2 that may dissolve in the condensate. Either denatured or absolute alcohol with an evaporation residue ≤ 0.001 percent by weight.

Filter. A flash-fired glass fibre filter (organic binder removed) of a diameter compatible with the filter holder with an efficiency of at least 99.95% for 0.3 µm particles in accordance with the most recent version of ASTM Standard D2986. The manufacturer's test data for the filter is sufficient. The filter material must be chemically inert to stack gas components such as sulphur dioxide (SO_2). Depending on the nature of the source and the analyses required, other types of filter media may be used, subject to approval by Environment Canada.

The filter must be desiccated to a constant weight before being used. This may be accomplished by desiccating the filter for at least 24 hours with silica gel or equivalent desiccant at 20 ± 6°C (68 ± 10°F). Weigh the filter to the nearest 0.1 mg at intervals of six hours or more in a room where the relative humidity is 50% or less. The weighing must be completed within two minutes after the filter is removed from the desiccator. The constant weigh is attained when the difference between two consecutive readings is less than 0.5 mg. Place the pre-weighed filter in a clean labeled Petri dish to prevent contamination during transportation to the sampling site.

Polymer Membrane Filter. The mist filter must be fibreless, have an efficiency of a least 99.95% for 0.3 micron particles, and have a 47 mm minimum diameter. The manufacturer's test data for the filter is sufficient. These filters do not need taring or final weighing.

Petri Dishes. Glass or plastic Petri dishes of a size greater than the filter. Each test run requires two Petri dishes: one for the glass fiber filter and another for the mist filter.

4.2 Sample Recovery

Water. As noted in Section 4.1.

Acetone. Reagent grade, for rinsing and sample recovery, shall have an evaporation residue ≤ 0.001 percent by weight.

Dichloromethane (DCM). Distilled-in-glass, for extracting CPM and rinsing, shall have an evaporation residue ≤ 0.001 percent by weight. Distilled-in-glass hexane may be substituted for DCM **only** when prohibited by the plant, province or territory.

> ***Caution: Precautions must be used when handing dichloromethane in the field. Ensure the recovery area is well-ventilated and use a respirator when handling this solvent. Use double layers of nitrile gloves when handling dichloromethane.***

Ammonium Hydroxide. 0.1 N NH_4OH volumetric solution that has been standardized against a NIST reference material, or equivalent.

Sample Jars. Chemically inert wide-mouth sample jars of a capacity of 250 mL to store the rinses of the nozzle, probe liner and the front half of the filter holder. For CPM, wide-mouth glass sample jars with a capacity compatible with the volume of the HPLC water and solvent rinses of the train components. Use tared 250 mL jars with the same supplier catalogue number for final gravimetric determination. Pre-labeled jars are recommended.

pH Indicator. Calibrated pH meter or pH indicator paper strips.

Extraction Vials and Sonicator. Clear vials with a capacity of 40 mL and a sonication bath able to deliver a frequency of 20 kHz.

Separatory Funnels. Borosilicate glass with a capacity of 500 mL to 2 L with a PTFE stopcock valve and cap.

Drying Oven. A vented temperature-controlled oven capable of controlling the temperature to 105°C (221°F) to within 3°C (5°F).

5. Procedures

5.1 Taring

Place the labeled sample jars in an oven and bake at 105°C for a minimum of six hours. Then transfer the jars to a desiccator containing silica gel or equivalent desiccant for a minimum of six hours.

Calibrate the balance and record the time, relative humidity, atmospheric pressure, the weight of a 200 g NBS Class "S" (or equivalent) weight and the reference jar and each jar of the set. One of the jars will serve as weight reference for the entire set. Correct the weight of each jar with respect to the reference jar (referenced weight).

The reference jar serves as an indicator of the buoyancy effect of the volume of glass in the jars. The buoyancy effect is dependent on temperature, barometric pressure and relative humidity, which affect the specific gravity of the air displaced by the glass in the jar. This will increase or decrease the apparent weight of a jar. The tracking of the reference jar apparent weight allows the correction for the buoyancy effect. The change in the reference jar apparent weight is used to correct the weight of the jars containing the residues.

After a minimum of six hours repeat the balance calibration, the ambient data recording and the weighing of the reference jar and each jar of the set. Correct the weight of each jar with respect to the reference jar (referenced weight).

If the consecutive (≥ 6 hr intervals) referenced weights are within 0.5 mg, then the final reading of the consecutive weights is the tare referenced weight of each jar. Otherwise, perform additional weighing until a constant referenced weight is achieved.

Review your weighing procedures if you are unable to achieve a 0.3 mg standard deviation for consecutive referenced weighing of empty 250 mL jars.

Cap and place each jar within a clean clear polyethylene bag for later use in the field. Keep the reference jar in the laboratory for later use as a reference while weighing the jars containing the sample residues.

Note: The same reference jar must be used for tare and residue referenced weights.

5.2 Sample Collection

Preliminary. In the absence of any previous knowledge of the stack variables, a preliminary test should be conducted to obtain the following data:

- Number and location of the traverse points spaced in accordance to Reference Method EPS1/RM/8 Method A.
- Velocity profile across the stack, stack temperature and pressure (Reference Method EPS 1/RM/8, Method B).
- Stack gas molecular weight (Reference Method EPS 1/RM/8, Method C).
- Stack gas moisture content (Reference Method EPS 1/RM/8, Method D).

All sampling train glassware shall be thoroughly cleaned in the laboratory prior to the test. A fresh set of glassware for the CPM portion of the train (condenser to the front-half mist filter) must be used for each run. No silicone grease shall be used to seal the glass joints subject to a DCM rinse. The glass joints must be sealed with fluoropolmer O-rings or Teflon tape.

Wash the glassware in a dishwasher with laboratory grade glass detergent. Some glass components such as the condenser and condensate trap may be cleaned by soaking overnight in a tub filled with water and detergent. After washing, rinse all glassware three times each with tap water, Type II water, acetone and finally with dichloromethane. The probe liner may be cleaned by brushing and rinsing the liner three times each with Type-II water and acetone. Cover the open ends of the glassware with cleaned aluminum foil.

Sampling Train Preparation and Operation. Assemble the sampling train in a clean area to minimize contamination. Install the selected size nozzle on the probe. Mark the probe with heat resistant markings to denote the location of each sampling point. Use a pair of tweezers to place the labeled and tared filter in the filter holder.

Sampling train preparation is similar to Method E, Reference Method EPS 1/RM/8, except for the installation of the mist filter and the addition of ethanol in the first stemless impinger or condensate trap to inhibit the oxidation of SO_2 in the condensate. Measure 3 mL of ethanol and make up to 10 mL with HPLC water and add to the condensate trap or stemless impinger. The first impinger contains 100 mL of HPLC water, the second impinger is left empty and the third one must contain 100 to 300 g of silica gel desiccant. The condensate/impinger setup is shown in Figure H-1. Record the weight of the impinger/condensate trap (after the ethanol addition), the condenser and the other three impingers to the nearest 0.5 g on the Moisture Analysis Data Sheet (Figure H-2)

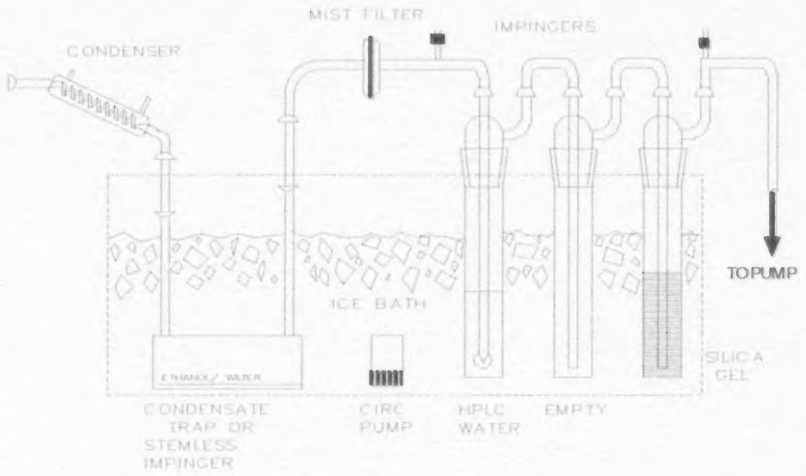


Figure H-1 Method H Impinger Train Schematic

Plant: _____
Location: _____
Test Number: _____
Date: _____
Weighing Conducted by: _____

Component	Contents	Weight (g)
Condenser Coil	Dry	Final _____ Initial _____ Gain _____ (a)
Condensate Trap pH = ___	3 mL ethanol made up to 10 mL with HPLC water	Final _____ Initial _____ Gain _____ (b)
First Impinger	100 mL HPLC	Final _____ Initial _____ Gain _____ (c)
Second Impinger	Empty	Final _____ Initial _____ Gain _____ (d)
Third Impinger	~ 200 g silica gel	Final _____ Initial _____ Gain _____ (e)
Total Moisture gain = a + b + c + d + e		_____ g

Figure H-2 Moisture Analysis Data Sheet

Follow the sampling procedures specified in Section 5.5.1 of Method E, Reference Method EPS 1/RM/8 for the collection of the train sample. These include the selection of the nozzle, pre- and post-test leak checks, sampling train operation and the recording of instrument readings.

5.3 Sample Recovery

Carefully disconnect the probe from the sampling train. Seal all openings. Exercise care in moving the train components from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter.

In the sample recovery area, weigh the components of the sample train. Record the weight to the nearest 0.5 g of the condenser, condensate trap and each of the three impingers on the Moisture Analysis Data Sheet (Figure H-2).

Recover the sample as follows:

Nozzle, Probe Liner and Front Half of Filter Holder Rinses (Containers 1 and 2). Carefully wipe any visible traces of particulate from the exterior surfaces of the nozzle and probe. Wash and brush the interior surfaces of the nozzle, probe liner and the front half of the filter holder with approximately 100 mL HPLC water. Store these rinsings in Container No.1. This container does not need to be tared. Then wash and brush the interior surfaces of the nozzle, probe liner and the front half of the filter holder with approximately 100 mL of acetone. Place these washings into Container No. 2. This container must be tared or quantitatively transferred to a tared container for the gravimetric determination. Seal and label the containers and mark the liquid levels. Use a fine tip marker to mark the levels on all the containers.

Primary Filter (Container 3). Use a pair of clean tweezers or a flat edge to transfer the glass fibre filter and loose material that may adhere to the filter support into a Petri dish. Seal and label the sample container

Condensate and Water Rinses (Container 4). Transfer the condensate collected in the condensate trap to Container 4. Measure and record the pH. Then rinse the primary filter back-half holder, condenser, condensate trap, connecting glassware and the front half of the mist filter holder three times each using a total of 100 mL HPLC water. Add the water rinses to Container 4. Use a second sample container (Container 4b) if the condensate and rinsing volumes exceeds the capacity of the jar. Fill the primary filter back-half holder and the condenser with HPLC water and allow to soak for five minutes. Add these soaks to Container 4. Seal and label the containers and mark the liquid levels.

Condenser and Back-half Rinses (Container 5). Fill the back half of the primary filter holder and the condenser coil with DCM and allow them to soak for five minutes. Add these soaks to Container 5. Then rinse the primary filter back-half holder, coil condenser, condensate trap, connecting glassware and the front half of the secondary filter three times each using a total of 100 mL DCM. Add these rinses to Container 5. Seal and label the container and mark the liquid level.

Secondary (Mist) Filter (Container 6). Use a pair of clean tweezers to place the filter into a Petri dish. Seal and label the container.

Front-half HPLC Water/Acetone Blank (Containers 7 and 8). Place 100 mL of water and 100 mL of acetone, each one taken directly from the corresponding wash bottle being used, into Containers No. 7 and 8, respectively. Container No. 7 does not need to be tared. Container No. 8 must be tared or quantitatively transferred to a tared container for the gravimetric determination. Seal and label the containers and mark the liquid levels.

Back-half HPLC Water/Ethanol Blank (Container 9). Take approximately 200 mL blank sample of HPLC water, and add three mL of ethanol, both used in the test runs, providing that the same stock solvent was used for all runs. Otherwise take separate blanks samples for each test run. Seal and label the container and mark the liquid level.

Dichloromethane Blank (Container 10). Take approximately 200 mL blank sample of DCM used in the test runs, providing that the same stock solvent was used for all runs. Otherwise take separate blanks samples for each test run. Seal and label the container and mark the liquid level.

Secondary (Mist) Filter Blank (Container 11). Use a pair of clean tweezers or a flat edge to place the filter into a Petri dish. Seal and label the sample container. This is required to confirm that no filter media material is being liberated into the water or DCM after sonication.

The use of tared glass containers is recommended to avoid sample transfer losses. During preparation and sample recovery these containers must be handled with laboratory gloves and protected from any alteration of their tare weight (additional labeling must be limited to the plastic cap). Following recovery, the containers are returned to their original polyethylene bags until their analysis in the laboratory. The containers must remain in an upright position at all times during shipping and maintained below 30°C.

5.4 Sample Analysis

All analyses shall be completed within two weeks of sampling in a clean laboratory equipped with a fume hood. The relative humidity of the room in which the weighing is performed should be maintained at or below 50%.

In the laboratory, remove the containers from their protecting polyethylene bags and verify that no liquid leaks had taken place. Leaks will invalidate the test results. Note the condition of the samples as received in the laboratory in Figure H-3.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Test Conducted by: _____

Condition of Samples as Received			
Container/ Jar No.		Description	Observations
1		Water and acetone rinses of nozzle, probe liner and front-half of the primary filter holder	Leakage during transport? Note condition of seal
2			Leakage during transport? Note condition of seal
3		Filter	Note condition
4		Condensate, soaks, HPLC rinses of back half of the primary filter holder, condenser, condensate trap, connecting glassware and front half of the mist filter	Leakage during transport? Note condition of seal
5		DCM soaks and rinses of back-half of the primary filter holder, condenser, condensate trap, connecting glassware and front half of the mist filter	Leakage during transport? Note condition of seal
6		Mist filter	Note condition
7		Front-half HPLC water/acetone blank	Leakage during transport? Note condition of seal
8			Leakage during transport? Note condition of seal
9		Back-half HPLC water/ethanol blank	Leakage during transport? Note condition of seal
10		Dichloromethane blank	Leakage during transport? Note condition of seal
11		Mist filter blank	Note condition

Figure H-3 Description of Received Samples

Sample analysis requires numerous gravimetric determinations of evaporation residues. Follow the procedures in Method G for the processing of Containers 1, 2, 3, 7 and 8. The following procedures are required for the determination of CPM. Refer to Figures H-4 and H-5. The samples in Containers 4 to 6 and 9 to 11 are required for the determination of CPM. Follow the sequence and procedures below for the determination of inorganic and organic condensable particulate matter.

Step	Procedure
1	Extract the water soluble CPM from the secondary filter (**Container 6**) by folding the filter in quarters and placing it into a 40-mL extraction tube. Add enough water to cover the secondary filter (approx. 10 to 20 mL). Place the tube into a sonication bath for a minimum of two minutes. Decant the aqueous extract into **Container 4** (or 4b, if used). Repeat this extraction step twice for a total of three extractions.
2	Extract the DCM soluble CPM from the water-extracted secondary filter (**Container 6**) by adding enough DCM to cover the filter (approx. 10 to 20 mL). Place the extraction tube into a sonication bath for a minimum of two minutes. Decant the DCM extract into **Container 5**. Repeat this extraction step twice for a total of three extractions.
3	Transfer contents of **Container 4** (and 4b, if used) to a separatory funnel. Then add 30 mL of DCM to the funnel, mix well and drain off the lower organic phase into **Container 5** Repeat twice with another 30 mL of DCM. This extraction should yield about 90 mL of organic extract. Each time, leave a small amount of DCM in the separatory funnel ensuring that no water is collected in the organic phase. Decant the aqueous phase back to **Containers 4** (or 4b, if used). ***Note: During the organic extraction process significant pressure may build up inside the separatory funnel. Make sure to relieve the pressure using the PTFE stopcock valve while mixing as a safety precaution.***
4	Evaporate the contents of the DCM extract **(Container 5)** at room temperature and pressure in a laboratory hood. Desiccate the residue for approximately 24 hours in a desiccator containing silica gel or equivalent desiccant. Weigh to a constant weight (± 0.5 mg) and report the results to the nearest 0.1 mg.
5	Place **Container 4** (and 4b, if used) in a 105°C (220°F) oven to evaporate the aqueous phase to not less than five mL. Then remove the container from the oven and complete the evaporation at room temperature and pressure in a desiccator containing silica gel or equivalent desiccant. Desiccate the residue for approximately 24 hours. Weigh to a constant weight (± 0.5 mg) and report the results to the nearest 0.1 mg. If a constant weight cannot be obtained on the residue contained in Container 4 (and 4b, if used) in four attempts then the presence of sulfuric acid in the sample is suspected, and the procedure in Step 6 must be followed.
6	Redissolve the residue with 100 mL HPLC water. Neutralize the acid in **Container 4** (and 4b, if used) by titration with 0.1 N NH_4OH solution. Determine the end point with a calibrated pH meter or a strip of pH indicating paper strip. Do not use liquid indicators, as even a few drops may add significant residue. Record the volume of titrant used. Again place **Container 4** (and 4b, if used) in a 105°C (220°F) oven to evaporate the aqueous phase to not less than 5 mL. Then remove the container from the oven and complete the evaporation at room temperature and pressure in a desiccator. Desiccate the residue for approximately 24 hours. Weigh to a constant weight (± 0.5 mg) and report the results to the nearest 0.1 mg. Subtract the weight of the ammonia contained in the titrant used by using the equation in Figure H-5.

Figure H-4 CPM Sample Processing

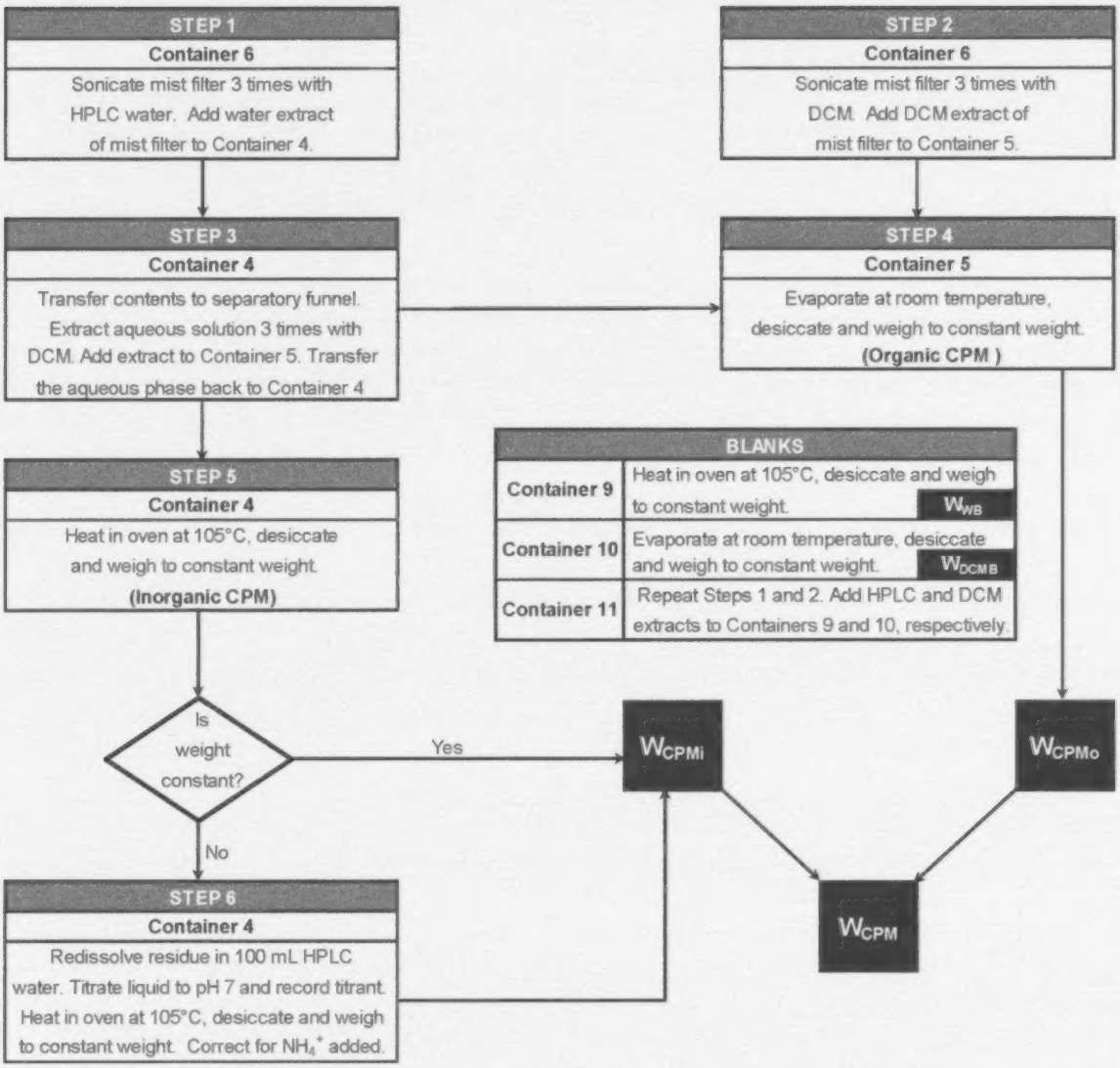

STEP 1
Container 6
Sonicate mist filter 3 times with HPLC water. Add water extract of mist filter to Container 4.
STEP 2
Container 6
Sonicate mist filter 3 times with DCM. Add DCM extract of mist filter to Container 5.
STEP 3
Container 4
Transfer contents to separatory funnel. Extract aqueous solution 3 times with DCM. Add extract to Container 5. Transfer the aqueous phase back to Container 4
STEP 4
Container 5
Evaporate at room temperature, desiccate and weigh to constant weight. (Organic CPM)
STEP 5
Container 4
Heat in oven at 105°C, desiccate and weigh to constant weight. (Inorganic CPM)
BLANKS
Container 9
Heat in oven at 105°C, desiccate and weigh to constant weight.
W_{WB}
Container 10
Evaporate at room temperature, desiccate and weigh to constant weight.
W_{DCMB}
Container 11
Repeat Steps 1 and 2. Add HPLC and DCM extracts to Containers 9 and 10, respectively.
Is weight constant?
Yes
No
W_{CPMi}
W_{CPMo}
W_{CPM}
STEP 6
Container 4
Redissolve residue in 100 mL HPLC water. Titrate liquid to pH 7 and record titrant. Heat in oven at 105°C, desiccate and weigh to constant weight. Correct for NH_4^+ added.

Figure H-5 CPM Sample Processing Flow Diagram

Residue Mass Determination. The determination of the evaporation residue may be affected by the weight and volume of the storage containers. The container weight and volume is typically six orders of magnitude greater than the residue. Therefore the taring and weighing may be affected by changes in air buoyancy.

Residue weighing should follow the procedure for taring the sample jars.

- Following the drying and desiccation of all samples, weight them and the reference container again.
- Calculate the loaded weights with respect to the final weight of the reference container.
- Weigh them again six or more hours later until the consecutive referenced weights differ by less than 0.5 mg
- The residue is the difference of the referenced final weight and the referenced tare weight of each sample.

Container 9 (Water/Ethanol Blank). Place **Container 9** (which also contains the aqueous extract of the mist filter blank) in a 105°C (220°F) oven to evaporate the aqueous phase to not less than 5 mL. Then remove the

container from the oven and complete the evaporation at room temperature and pressure. Desiccate the residue for approximately 24 hours in a desiccator containing silica gel or equivalent desiccant. Weigh to a constant weight and report the results to the nearest 0.1 mg.

Container 10 (DCM Blank). Evaporate the contents of the DCM (**Container 10**) (which also contains the DCM extract of the mist filter blank) at room temperature and pressure in a laboratory hood. Desiccate the residue for approximately 24 hours in a desiccator containing silica gel or equivalent desiccant. Weigh to a constant weight and report the results to the nearest 0.1 mg.

Record the weights in Figure H-6. Use the calculation in Figure H-6 to correct for the mass of NH_4^+ added to the residue during the titration.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Test Conducted by: _____

Train Component	Description	Final Weight (mg)	Tare Weight (mg)	Weight of Particulate (mg)	Fraction
Container 2 Container 3	Probe residue Primary filter			(a) (b)	
Container 8	Water/Acetone blank			(c)	
Container 4	Inorganic CPM			(d)	W_{CPMi}
Container 5	Organic CPM			(e)	W_{CPMo}
Container 9	Water blank			(f)	W_{WB}
Container 10	DCM blank			(g)	W_{DCMB}
If the inorganic residue was titrated with NH_4OH, calculate the mass added to the residue as shown below.					
Volume of Titrant (V_t) NH_4OH Normality Mass Correction for NH_4^+	____ mL 0.1 N $W_{titrant} = 17.03 \times V_t \times 0.1$			(h)	$W_{titrant}$

PM	=	$W_{PM} = (a - c) + b$ W_{PM} = _____ mg
CPM	=	$W_{CPM} = (d - f) + (e - g) - h$ W_{CPM} = _____ mg
TPM	=	$W_{PM} + W_{CPM}$ W_{TPM} = _____ mg

Notes:

- *The weight of the blanks must not exceed 2 mg*
- *Do not correct for negative blank values*
- *No blank corrections are allowed when blank residue is above 2 mg*

Figure H-6 Particulate Analytical Data Sheet

6. Calculations

During a test run, field data should be entered in the units of the stack sampling equipment (Imperial or common practice metric units). The equations of this method follow the common practice units, to facilitate its application and to avoid errors. The results may be converted, if necessary, to the units specified in the applicable standards or guidelines. The use of term weight actually refers to mass. The abbreviation "W" is used for weight to avoid confusion with "M" which is used for molecular weight.

Calculate the stack gas parameters as per section 5.6 of Method E, Reference Method EPS 1/RM/8.

The method detection limit for the gravimetric determination of residues from jars is 0.42 mg. The total expanded uncertainty (95% probability) for filterable PM and condensable particulate matter is 1.4 mg. The inorganic CPM data from nine paired runs at industrial source was used to compare this method to EPA Method 202. A statistical analysis of the differences between the two methods yielded a t-value of 0.28 with an average difference of -0.16 mg (95% confidence interval -1.5 to 1.1 mg).

Concentration of PM or CPM or TPM. Calculate the concentration of using Equation H-1

$$C_{PM} \text{ or } C_{CPM} \text{ or } C_{TPM} = \frac{W_{PM} \text{ or } W_{CPM} \text{ or } W_{TPM}}{(V_m)_{ref}} \qquad \textbf{Equation H-1}$$

Mass Emission Rate. Calculate the mass emission or release rate of PM, CPM or TPM using Equation H-3.

$$ER_{PM} \text{ or } ER_{CPM} \text{ or } ER_{TPM} = 10^{-6}(C_{PM} \text{ or } C_{CPM} \text{ or } ER_{TPM})Q_s \qquad \textbf{Equation H-2}$$

7. Nomenclature

C_{PM}	– concentration of filterable particulate matter in the stack gas on a dry basis at reference temperature and pressure, mg/m^3
C_{CPM}	– concentration of condensable particulate matter in the stack gas on a dry basis at reference temperature and pressure, mg/m^3
C_{TPM}	– concentration of total particulate matter in the stack gas on a dry basis at reference temperature and pressure, mg/m^3
ER_{PM}	– mass emission rate of filterable particulate matter, kg/h
ER_{CPM}	– mass emission rate of condensable particulate matter, kg/h
ER_{TPM}	– mass emission rate of total particulate matter, kg/h
Q_s	– volumetric stack gas flow rate on a dry basis at reference temperature and pressure, m^3/h
$(V_m)_{ref}$	– volume of stack gas sample on a dry basis at reference temperature and pressure, m^3
W_{PM}	– weight of filterable particulate collected during the test run, mg
W_{CPM}	– weight of condensable particulate collected during the test run, mg
W_{TPM}	– weight of total particulate collected during the test run, mg
10^{-6}	– conversion, kg/mg

Method I: Options

1. Applicability

This method is used to measure the mass concentration and mass emission or release of filterable $PM_{2.5}$ and condensable particulate matter (CPM) released from stationary sources. The front-half of Method G is combined with the back-half of Method H to form a method for fine particulate matter from stationary sources. The sum of the filterable $PM_{2.5}$ and the condensable particulate matter will give total $PM_{2.5}$. This method is subject to the limitations of Methods G and H.

2. Principle

The principles provided in Methods G and H are applicable to this method. Three valid tests are required for the determination of total $PM_{2.5}$. Each test must be a minimum of two hours and collect at least 1.5 m^3 (53 ft^3) of stack gas on a dry basis at reference conditions.

3. Apparatus

As per Methods G and H.

4. Reagents and Materials

The reagents and materials listed in Section 4 of Method H must be utilized when combining Methods G and H. This includes the use of HPLC grade water used for rinsing both the front- and back-half components.

5. Procedures

5.1 Taring

As per Method H.

5.2 Sample Collection

Sample Rate Selection. Use the procedures in Section 5.1 and Figure G-9 of Method G to select an appropriate sampling rate for the cyclone such that the criteria for dwell times, cutoff diameter and isokinetics are met. Select a sample run time greater than two hours that results in a sample volume of at least 1.5 m^3 (53 ft^3) at dry standard conditions.

Sample Train Preparation. Combine the front half of the Method G train with the back half of the Method H train as depicted in Figure I-1. Measure 3 mL of ethanol and make up to 10 mL with HPLC water and add to the condensate trap or stemless impinger. The first impinger contains 100 mL of HPLC water, the second impinger is left empty and the third one must contain 100 to 300 g of silica gel desiccant. Record the weight of the impinger/condensate trap (after the ethanol addition), the condenser and the other three impingers (to the nearest 0.5 g) on the Moisture Analysis Data Sheet (Figure H-2).

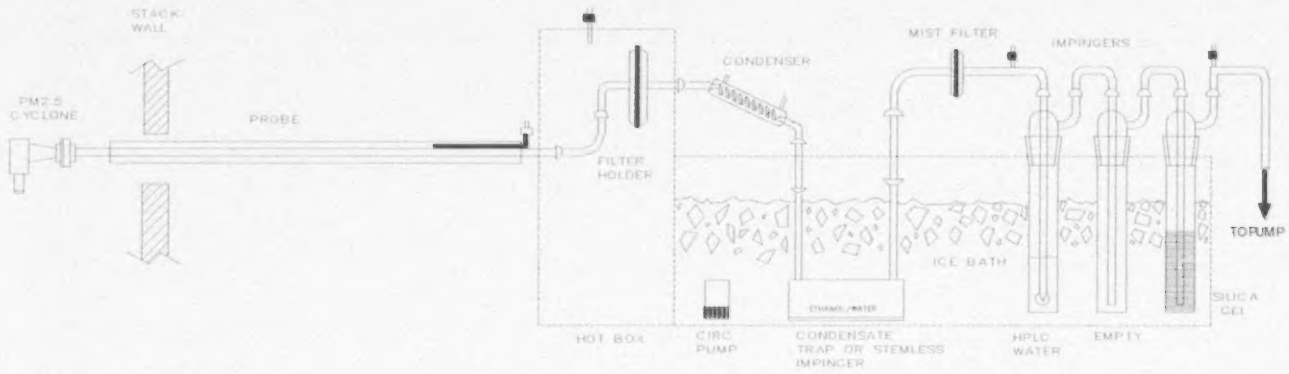


Figure I-1 Method I Schematic: Combined Methods G and H

Sample Train Operation. As per Method G, use Figure G-9 or a similar strategy to determine the orifice setting at each sampling point and a sample time resulting in a total sample volume of at least 1.5 m^3 (53 ft^3) at dry standard conditions.

5.3 Sample Recovery

Carefully disconnect the probe from the sampling train, seal all openings. Seal the openings of the $PM_{2.5}$ cyclone. Exercise care in moving the train components from the test site to the sample recovery area to minimize the loss of collected particulate matter or contamination.

In the sample recovery area, weigh the components of the sample train. Record the weight (to the nearest 0.5 g) of the condenser, condensate trap and each of the three impingers on the Moisture Analysis Data Sheet (Figure H-2).

Recover the front-half train components as described in Section 5.2 of Method G using HPLC water in place of Type II to avoid accidental use of Type II water in the CPM recovery. Recover the back-half train components as described in Section 5.3 of Method H.

5.4 Sample Analysis

All analyses shall be completed within two weeks of sampling in a clean laboratory equipped with a fume hood. The relative humidity of the room in which the weighing is performed should be maintained at or below 50%.

In the laboratory, remove the containers from their protecting polyethylene bags and verify that no liquid leaks had taken place. Leaks will invalidate the test results. Note the condition of the samples as received in the laboratory in Figure I-2.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Test Conducted by: _____

Condition of Samples As Received			
Container/ Jar No.		**Description**	**Observations**
1		Water and acetone rinses of cyclone and nozzle (combine as per Method G)	Leakage during transport? Note condition of seal
2		Water and acetone rinses of cyclone exit tube, probe liner and front half of the primary filter holder (combine as per Method G)	Leakage during transport? Note condition of seal
3		Filter	Note condition
4		Condensate, soaks, HPLC rinses of back half of the primary filter holder, condenser, connecting glassware and front half of the mist filter	Leakage during transport? Note condition of seal
5		DCM soaks and rinses of back half of the primary filter holder, condenser, connecting glassware and front half of the mist filter	Leakage during transport? Note condition of seal
6		Mist filter	Note condition
7		Front-half HPLC water/acetone blank	Leakage during transport? Note condition of seal
8			Leakage during transport? Note condition of seal
9		Back-half HPLC water/ethanol blank	Leakage during transport? Note condition of seal
10		Dichloromethane blank	Leakage during transport? Note condition of seal
11		Mist filter blank	Note condition

Figure I-2 Description of Received Samples

Sample analysis requires numerous gravimetric determinations of evaporated residues. Follow the procedures in Method G for processing Containers 1, 2, 3, 7 and 8. Follow the procedures in Method H for Containers 4, 5, 6, 9, 10 and 11. Once a constant weight has been determined for all the sample residues use the Particulate Analytical Data Sheet in Figure I-3 to determine the weight of the different particulate fractions.

Plant: _____
Location: _____
Test Number: _____
Date: _____
Test Conducted by: _____

Train Component	Description	Final Weight (mg)	Tare Weight (mg)	Weight of Particulate (mg)	Fraction
Container 1	+$PM_{2.5}$			(a)	
Container 2	$PM_{2.5}$			(b)	
Container 3	Primary filter			(c)	
Container 8	Water/Acetone blank			(d)	
Container 4	Inorganic CPM			(e)	W_{CPMi}
Container 5	Organic CPM			(f)	W_{CPMo}
Container 7	Water blank			(g)	W_{WB}
Container 9	DCM blank			(h)	W_{DCMB}
If the inorganic residue was titrated with NH_4OH, calculate the mass added to the residue as shown below.					
Volume of Titrant (V_t) NH_4OH Normality Mass Correction for NH_4^+	____ mL 0.1 N $W_{titrant} = 17.03 \times V_t \times 0.1$			(i)	$W_{titrant}$

Filterable $PM_{2.5}$	=	$W_{PM2.5} = (b - d) + c$ $W_{PM2.5}$ = _____ mg
CPM	=	$W_{CPM} = (e - g) + (f - h) - i$ W_{CPM} = _____ mg
Total $PM_{2.5}$	=	$W_{TPM2.5} = W_{PM2.5} + W_{CPM}$ $W_{TPM2.5}$ = _____ mg
Filterable PM	=	$W_{PM} = a + (b - d) + c$ W_{PM} = _____ mg
Total PM	=	$W_{TPM} = W_{PM} + W_{CPM}$ W_{TPM} = _____ mg

Notes:

- *The weight of the blanks must not exceed 2 mg*
- *Do not correct for negative blank values*
- *No blank corrections are allowed when blank residue is above 2 mg*

Figure I-3 Particulate Analytical Data Sheet

6. Calculations

Calculate the stack gas parameters as per Section 6 of Method G. If applicable, calculate the filterable PM, $PM_{2.5}$ and CPM concentrations and emission rates as per Section 6 of Methods G and H.

The method detection limit for the gravimetric determination of residues from jars is 0.42 mg. The total expanded uncertainty (95% probability) for TPM, that consists of filterable PM (includes +$PM_{2.5}$ and $PM_{2.5}$) and condensable particulate matter, is 1.5 mg.

Concentration of TPM or $TPM_{2.5}$. Calculate the concentration of TPM or $TPM_{2.5}$ using Equation I-1

$$C_{TPM} \text{ or } C_{TPM\,2.5} = \frac{W_{TPM} \text{ or } W_{TPM\,2.5}}{(V_m)_{ref}} \qquad \textbf{Equation H-1}$$

Mass Emission Rate. Calculate the mass emission or release rate of TPM or $TPM_{2.5}$ using Equation H-2.

$$ER_{TPM} \text{ or } ER_{TPM\,2.5} = 10^{-6}\,(C_{TPM} \text{ or } C_{TPM\,2.5})\,Q_s \qquad \textbf{Equation H-2}$$

7. Nomenclature

$C_{TPM2.5}$	– concentration of total $PM_{2.5}$ in the stack gas on a dry basis at reference temperature and pressure, mg/m^3
C_{TPM}	– concentration of total particulate matter in the stack gas on a dry basis at reference temperature and pressure, mg/m^3
$ER_{TPM2.5}$	– mass emission rate of total $PM_{2.5}$, kg/h
ER_{TPM}	– mass emission rate of total particulate matter, kg/h
Q_s	– volumetric stack gas flow rate on a dry basis at reference temperature and pressure, m^3/h
$(V_m)_{ref}$	– volume of stack gas sample on a dry basis at reference temperature and pressure, m^3
$W_{TPM2.5}$	– weight of total $PM_{2.5}$ collected during the test run, mg
W_{TPM}	– weight of total particulate collected during the test run, mg
10^{-6}	– conversion, kg/mg

References

Environment Canada, *Reference Method for Source Testing: Measurement of Releases of Particulate from Stationary Sources*, Reference Method EPS 1/RM/8, December 1993.

U.S. Environmental Protection Agency. Federal Reference Methods 201a and 202. *Methods for Measurements of Filterable PM_{10} and $PM_{2.5}$ and Measurement of Condensable PM Emissions from Stationary Sources,* 40 CFR Part 51.

U.S. Environmental Protection Agency. Federal Reference Method 30b, *Determination of Mercury from Coal-Fired Combustion Sources Using Carbon Sorbent Traps,* 40 CFR Part 60, Appendix A-8

Wang, L., Y. Ma, J. Hao and Y. Zhao. *Mechanism and Kinetics of Sulfite Oxidation in the Presence of Ethanol.* Ind. Eng. Chem. Res. 48, 4307-4311, 2009.

www.ec.gc.ca

Additional information can be obtained at:

Environment Canada
Inquiry Centre
10 Wellington Street, 23rd Floor
Gatineau QC K1A 0H3
Telephone: 1-800-668-6767 (in Canada only) or 819-997-2800
Fax: 819-994-1412
TTY: 819-994-0736
Email: enviroinfo@ec.gc.ca